



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

74th Starch Convention

April 18th – 19th 2023

program
supporting program
list of participants
summaries

Tuesday, April 18th 2023

08⁰⁰ – 08³⁰ Registration

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

1. Market

08⁴⁵ 1.1. **Jamie Fortescue**, Brussels (Belgium)
Current political/regulatory priorities for EU starch producers

2. Starch properties and modification

09¹⁵ 2.1. **Oswaldo H. Campanella**, Columbus (USA)
The creation of functional structures in starches and starch/non-starch polysaccharides mixtures by the application of controlled shear forces

09⁴⁵ 2.2. **Marcus Schmidt**, Detmold (Germany)
Genotype-specific starch characteristics in relation to resistant starch formation in table potatoes

10¹⁵ 2.3. **Jens Buller**, Berlin (Germany)
Chemoselective oxidation of C6 primary hydroxyl groups in starch - a critical analysis of the potentials

10⁴⁵ **Communication Break**

11¹⁵ 2.4. **D.C. Saxena**, Punjab (India)
Studies on synthesis of nanoparticles from Guinea grass seed (*Megathyrsus maximus*) starch for development of Pickering emulsions

11⁴⁵ 2.5. **Hans Leemhuis**, Groningen (Netherlands)
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12¹⁵ 2.6. **Guillermo Portillo**, Aarhus (Denmark)
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12⁴⁵ **Lunch Break**

3. Application

14⁰⁰ 3.1. **Florian Wurm**, Innsbruck (Austria)
The application of cationic starch as an auxiliary in reactive textile dyeing

4. Food / pharmaceutical applications

14³⁰ 4.1. **Robert Gilbert**, Queensland (Australia) **-ONLINE-**
Towards rice which is both healthier and tastier:
how endogenous proteins affect starch digestion in grain-based foods

15⁰⁰ 4.2. **Janusz Kapusniak**, Czestochowa (Poland)
Fiber and prebiotic substances from starch in human nutrition

15³⁰ **Communication Break**

16⁰⁰ 4.3. **Tim Terstegen**, Berlin (Germany)
Organic pea starch – Optimization for the application in food matrices

16³⁰ 4.4. **Jovin Hasjim**, Lestrem (France)
Roquette's journey in the development of glycemic carbohydrates with slow digestion

5. Future developments

17⁰⁰ 5.1. **Nelli Elizarov**, Berlin (Germany)
Sustainability in Transport through Renewable Fuels

17³⁰ 5.2. **Arjen Postma, Haarlem** (Netherlands)
New BioSolutions based on LpHera technology helps you to achieve more sustainable liquefaction, reduce energy and CO2 footprint

Wednesday, April 19th 2023

6. Technology

08³⁰ 6.1. **Vijay Singh**, Urbana (USA)
Characterization and comparison of wet milling fractions of export commodity corn originating from different international geographical locations

09⁰⁰ 6.2. **Ted Slaghek**, Wageningen (Netherlands)
Electrification of the starch oxidation process

09³⁰ Communication Break

10⁰⁰ 6.3. **Maurice Essers**, Wageningen (Netherlands)
Hydrophobic starches via reactive extrusion

10³⁰ 6.4. **Martin Heckl**, Freising (Germany)
Characterisation of starch granules with different analytical methods for use in printing materials for food 3D printing

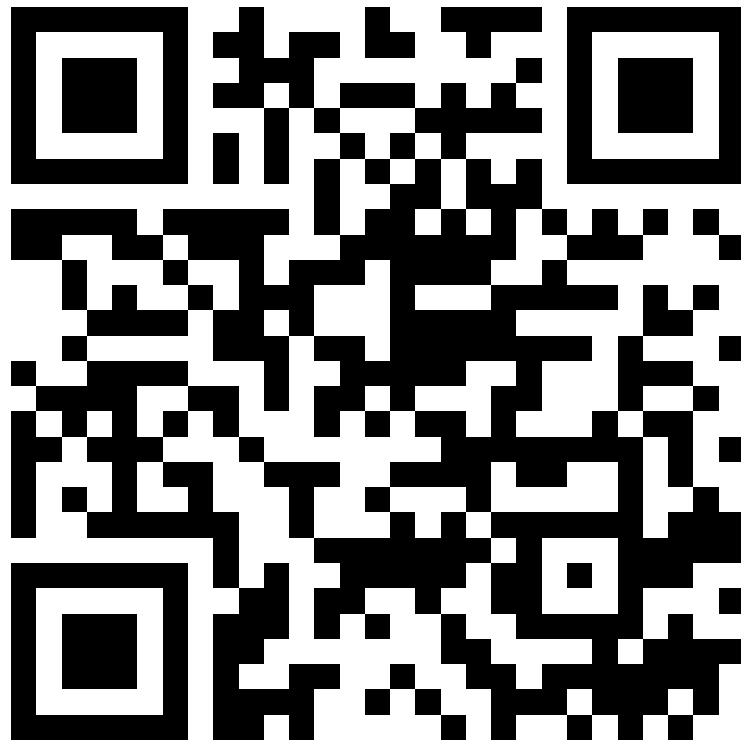
11⁰⁰ 6.5. **Lubbert Dijkhuizen**, Groningen (Netherlands)
Novel alpha-Glucanotransferase enzymes that introduce alpha1-6 or alpha1-3 linkages in starch, yielding soluble dietary fibers

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

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Lunch

Lunch will be served in the exhibition hall:

The menu:

Curry mango chicken with coconut milk, tomato and rice

Malysian chickpea curry, vegan with tomato, coconut milk, red lenses

Potato in 3 different tastes:

As rubbing cookies with salmon and yogurt dip

Potato salad with green sauce, vegetarian and with meatballs

Mashed potatoes with different toppings, with cheese / bacon / sweet pepper

Sweet potato tortilla, vegetarian and with chorizo

Brioche braid with wild garlic and sun-dried tomatoes

Tangerine curd and fruit salad

Beverages:

Mineral water

Coca-Cola

Orange juice

Apple Spritzer

**Bon appétit
and interesting conversations!**

Evening Program

Monday, April 17th 2023

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

The menu at the welcome evening:

Chopped chicken with mushrooms, courgetti and sweet pepper

Redfish in mustard sauce

Vegetarian pasta with Mediterranean vegetables

Side dishes:

Basmati rice

Potato gratin

Mixed lettuce, colourful vegetable selection

Panna Cotta with raspberry sauce & Mousse au chocolate

Tuesday, April 18th 2023

19⁰⁰ **“Bread and Beer tasting”- Get-together** in the “Haus des Brotes” (Exhibition Hall), Detmold, Schuetzenberg 10

Participants

Effective April 13th, 2023, 04.00 p.m.

Abdelfattah, Islam Mohamed Gharieb Abeln, Dieter	Egyptian Starch and Glucose Co. (Egypt) Behn & Bates Maschinenfabrik GmbH & Co. KG, Münster
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Bischoff, Felix, Dr.	Jäckering Mühlen- und Nahrungsmittelwerke GmbH, Hamm
Brinkmann, Franz Broekman, Onno Brümmer, Thomas, Dr.	Gebr. Lödige Maschinenbau GmbH, Paderborn University of Groningen (The Netherlands) Brümmer Extrusion Consulting, Wittenbach (Switzerland)
Buller, Jens, Dr.	Fraunhofer Institut für Angewandte Polymerforschung, Potsdam-Golm
Busch, Peter Campanella, Osvaldo H., Prof.	Levaco Chemicals GmbH, Leverkusen Carl E. Haas Endowed Chair in Food Industries, Columbus (USA)
Christensen, Stefan Jarl	KMC Kartoffelmelcentralen a.m.b.a., Brande (Denmark)
Dijkhuizen, Lubbert, Prof. Dr.	CarbExplore Research BV, AN Groningen (The Netherlands)
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Khidr, Raafat Mohamed Aly	National Company for Maize Products (Egypt)

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Krogt, van der, Do	Royal Avebe u. A., Veendam (The Netherlands)
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Gustavo, Prof.	Biotecnología, Quito (Ecuador)
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Koch, Maximilian, Dr.
Langenkämper, Georg, Dr.
Matthäus, Bertrand, Dr.
Meissner, Philipp, Dr.
Meyer, Christina, Dr.
N'Diaye, Katharina (wiss. Mitarbeiterin)
Nikolay, Sharline, (M.Sc.)
Schmidt, Marcus, Dr.
Schubert, Madline, Dr.

Schuster, Ralph (Dipl. Ing. (FH))
Scheibner, Andreas
Schwake-Anduschus, Christine, Dr.
Sciurba, Elisabeth, Dr.
Sieren, Theresa (M.Sc.)
Smit, Inga, Dr.
Stake, Kirsten
Thüm, Marcus
Themeier, Heinz, Dipl.-Ing.
Unbehend, Günter, Dipl.-Ing.
Vosmann, Klaus, Dr.
Weber, Lydia, Dipl.oec.troph.
Wolf, Klaus

Summaries

1. Market

- 1.1. **Jamie Fortescue**, Brussels (Belgium)
Current political/regulatory priorities for EU starch producers

When the current European Parliament and Commission took office in 2019, an ambitious Green Deal Agenda was announced, followed by a series of plans notably 'The Farm to Fork Strategy' and the 'Fit for 55 package', all designed to list the concrete actions/proposals that would be delivered by 2024, before the next Commission and Parliament took office. Then there was COVID and then there was Russian invasion of Ukraine. Jamie will provide an overview of what progress has been made on the Green Deal agenda, what may still come before 2024, what is likely to be delayed and how and how all this may impact EU starch producers.



Jamie Fortescue, British/Belgian, born 1966, joined the European Starch Industry Association, Starch Europe, in January 2012 as its managing director. Supported by a team of six staff, his role is to represent the interests of EU starch producers to the EU institutions and other EU stakeholders. From 2014-2011, Jamie was the Director General of Spirits Europe, the trade association representing EU spirits drinks producers. Prior to that, he was the director for international corporate affairs at the employment agency, Randstad, based in Amsterdam (1997-2004) and an account director at the public affairs consultancy, EPPA, based mainly from their offices in London and Brussels, but also Madrid and Warsaw (1992-1997). Working primarily on issues affecting the petfood and employment agency sectors. Before becoming involved in EU public affairs, Jamie worked in third world development (at London based Planco Consulting from 1989-91, and as a trainee at DG Development of the European Commission from 1988-89). Jamie was educated at Eton College and the University of East Anglia, and has a degree in French and Economics.

2. Starch properties and modification

- 2.1. **Oswaldo H. Campanella**, Columbus (USA)
The creation of functional structures in starches and starch/non-starch polysaccharides mixtures by the application of controlled shear forces

Starch is the most common energy source in human nutrition and has been widely used as gelling and thickening agent in many food applications. However, and despite these benefits, some controversial information has reduced its use in the production of foods, and it has shifted to the use on non-food products. Disadvantages of the use of starch for food applications have focused on health concerns including postprandial blood glucose spikes, high Glycemic Index (GI) and the use of chemicals to improve starch functionality to produce foods. These concerns has forced scientists to search for possibilities of getting stable structures that reduce these health concerns by using physical means and mixing with other food components (e.g., fibers and proteins) to enhance the food texture while at the same improving its nutritional properties.

Concerning physical means to modify starches shear forces are important and they can be controlled to evaluate their effects on product quality. However, there are not many studies on shear-induced structure changes of starch and starch/hydrocolloid systems. It has shown that gelatinized waxy potato starch forms strong viscoelastic gels under the application of long-term low shear at low temperatures, a phenomenon that have not been noted in other waxy starches such as rice and corn. Other studies have shown that the viscoelastic properties of gelatinized waxy potato starch in the presence of neutral (guar gum and konjac glucomannan) and anionic hydrocolloids (gum arabic, xanthan gum, sodium alginate, and pectin) were significantly enhanced when gelatinized waxy potato starch/neutral hydrocolloid dispersions where sheared at 20 s^{-1} for 24 h at $5 \text{ }^{\circ}\text{C}$.

Concerns about food security and healthy diets have shifted the focus from traditional meat products toward plant protein-based meat analogues (MA), which contain a large protein content, negligible saturated fat, and cholesterol. Proteins in MAs form a dense mesh with aligned fibrils

promoted by thermal–mechanical treatments in an extruder or high-pressure shears cell providing their unique viscoelastic properties. The use of large content of protein, preferably isolates, to achieve a desired texture has a large incidence in the cost of these products. Recent studies have shown that starch and fibers are potential ingredients to reduce the ingredient cost, achieve good texture and provide the energy delivered by starches. Work has shown that the addition of fibrils formed by heating whey protein isolate (WPI) under different pHs to gelatinized potato starch promoted the associative interactions between fibrils fabricated at acid pH and starch to synergistic increases gel elasticity but not those formed at neutral pH conditions under which starch and protein were poorly compatible. Similar results were obtained when pectin was added to pea protein isolate, which is commonly used in production plant protein-based meat products. Furthermore, rice starch was used as a key component to produce plant-based proteins with textures like a variety of meat, fish and dairy products. These results indicate that starch and fiber could be key components in the production of these plant-based analogues.

Mechanical work through extrusion processes under especial shear conditions can produce molecular scissions that promote interactions resulting in slowly digestible foods. Results suggest that the nature of the structurally driven slowly digestible starch is mostly due to molecular interactions involving amylopectin, which do not necessarily worsen the food mechanical properties.

Results of these processing/formulation options to promote the use of starch in the production of foods will be discussed during the presentation.



Professor Osvaldo Campanella is the Carl E. Haas Endowed Chair in Food Industries at the Ohio State University since 2019. Previously he was a professor at Purdue University in the Agricultural and Biological Engineering Department and the Whistler Carbohydrate Research Center since 1999, position he holds as emeritus professor.

2.2. **Marcus Schmidt**, Detmold (Germany)

Genotype-specific starch characteristics in relation to resistant starch formation in table potatoes

Marcus Schmidt, Lydia Weber, Jens Begemann, Inga Smit

Max Rubner-Institut, Federal Research Institute of Nutrition and Food, Department of Safety and Quality of Cereals, Schützenberg 12, 32756 Detmold

Potatoes are important staple foods, rich in carbohydrates but also in minerals and vitamins. In Germany, a share of 36% of potatoes are table potatoes, which are consumed after cooking. Table potatoes play an important role as carbohydrate-rich side dishes, since they reach a higher satiety index despite lower energy intake compared to rice or pasta. However, since they also present a higher glycaemic index (GI), contradicting dietary recommendations are given. Previous studies identified the content of resistant starch (RS), formed due to retrogradation of gelatinized starch after cooking, as primary criterium for the GI. However, there are substantial differences between varieties regarding the speed of retrogradation and the total amount of RS formed. It is assumed, that these variations depend on the genotype-specific molecular characteristics of the potato starch. Hence, it was the aim of this study to investigate the starch molecular properties in relation to the formation of RS.

Six common table potato varieties (Huckleberry Gold, Goldmarie, Marry Ann, Soraya, Adretta, Talent), including one with reportedly low GI, were used to investigate the starch pasting and digestibility in whole tubers and isolated starches. A Micro-Visco Amylograph was used to obtain pastes of the isolated starches and determine the pasting curves. *In vitro* starch digestibility was determined for raw freeze-dried tubers, cooked tubers kept at 4°C for up to 72h and isolated pasted starches. Moreover, important molecular properties, including granule size distribution, molar mass distribution, amylose content and inter- and intra-molecular structures, were determined for the isolated starches.

The results show substantial differences in starch digestibility among genotypes. Immediately after cooking Huckleberry Gold contained lowest content of rapidly digestible starch (RDS), but showed slower decrease during 72 h of cold storage compared to other varieties. Soraya starch had small and low-branched amylopectin alongside with small granule size as characteristics for rapid RS formation in isolated starch. However, in the whole tuber, Soraya showed the least RS formation within 24 h after cooking. The contrary behaviour was found for Huckleberry Gold, which formed RS in the tuber already shortly after cooking, whereas slow RS formation was evident in the isolated starch. The results suggest, that starch structural characteristics play a role in RS formation but are not sufficient to fully explain the starch retrogradation in the tuber.

Nutritional characteristics, such as the formation of RS can be improved by suitable starch molecular characteristics. Therefore, starch characteristics might be promising as complementary and specific breeding aspects for varieties with low GI and high nutritional value. However, additional factors, such as non-starch constituents have to be considered also but need further research.



Dr. Marcus Schmidt studied food chemistry at the Technical University of Dresden, Germany, graduating with a diploma. He conducted research on bio-preservation of cereals and cereal products with a PhD in Food Science and Technology at University College Cork (UCC), Ireland under the supervision of Prof. Elke Arendt. Since 2020, he is working at the Max Rubner-Institut as head of the Carbohydrate Analysis group. His current research interests include work on the production of low-FODMAP (fermentable oligo-, di-, and monosaccharides, as well as polyols) baked goods, the health-promoting properties of dietary fiber, increased food security through utilization of side-streams and the carbohydrate composition of potatoes.

2.3. **Jens Buller**, Berlin (Germany)

Chemoselective oxidation of C6 primary hydroxyl groups in starch - a critical analysis of the potentials

Buller J. *, Städtke K., Gabriel-Liebs C., Wetzel H.

Fraunhofer Institute for Applied Polymer Research, Department of Starch Modification / Molecular Properties, Germany

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The selective oxidation of cellulose, via oxidation catalysis with oxoammonium salts (2,2,6,6-tetramethylpiperidinyloxy, TEMPO) has been well known for some years [1]. For starch, on the other hand, there are still few publications. For the functionalization of corn and waxy corn starches the regioselective TEMPO oxidation of C6 primary hydroxyls [2] was thus applied in aqueous slurry and paste conditions. Several TEMPO derivatives and experimental parameters have been tested to achieve high degrees of functionalization (oxidation). The number of carboxyl groups generated, was determined and different analytical methods evaluated for the analysis of carboxyl and carbonyl groups. Namely FTIR, HPAE-PAD, titration, or ICP-OES. Additional NMR measurements confirmed the selective oxidation at C6. The molar mass of the oxidized starches was investigated by GPC-MALS. Under the selected synthesis conditions with NaOCl as oxidant, a maximum of about 70 carboxyl groups per 100 anhydroglucose units was generated. However, a high use of oxidizing agent also showed a substantial decrease in molar mass to about $2 \cdot 10^5$ g/mol.

Milder conditions were used for the selective oxidation of starch with the aid of atmospheric oxygen and the enzyme laccase as a co-catalyst. With these sustainable reaction conditions, functionalization levels up to 20 carboxyl groups per 100 anhydroglucose units could be achieved in the optimal laccase temperature range of 70 °C. Thereby, only a minor molecular mass degradation took place.

All in all, chemoselective oxidation can be an interesting method for the synthesis of new products from starch. Especially the good degradability of the polyuronic acids, which was shown in degradability tests, can be advantageous compared to other functionalized starches. However, further work is needed to improve the reaction with regard to the use of toxic substances, the efficiency, the reaction conditions or the economic use of the catalyst for commercial exploitation.

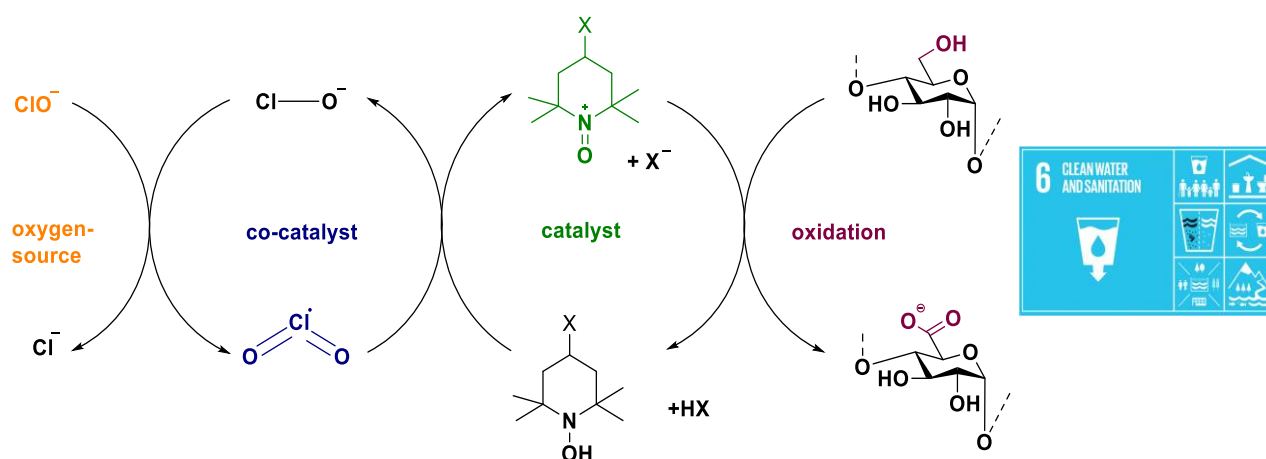


Figure 1 : Mechanism of starch oxidation by TEMPO/NaClO/NaClO₂

[1] Pierre, Guillaume, et al. "TEMPO-mediated oxidation of polysaccharides: An ongoing story." *Carbohydrate polymers* 165 (2017): 71-85.

[2] Bobbitt, James M. "Oxoammonium Salts. 6.1 4-Acetylamino-2, 2, 6, 6-tetramethylpiperidine-1-oxoammonium perchlorate: a stable and convenient reagent for the oxidation of alcohols. silica gel catalysis." *The Journal of Organic Chemistry* 63.25 (1998): 9367-9374.



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.

2.4. D.C. Saxena, Punjab (India)

Studies on synthesis of nanoparticles from Guinea grass seed (*Megathyrsus maximus*) starch for development of Pickering emulsions

In the present study, the unconventional Guinea seed starch was isolated, characterized, and used to prepare starch nanoparticles via the nanoprecipitation method. Guinea starch was isolated using three different methods: sodium hydroxide (NaOH), citric acid (CA), and distilled water (DW). The water absorption capacity (WAC) and oil absorption capacity (OAC) of guinea starch ranged from 0.69 to 1.12 g/g and 0.81 to 0.95 g/g, respectively. The swelling and solubility of isolated starches were temperature-dependent and increased with temperature (70 to 90 °C). The amylose content varied from 28.18% to 25.69%, depending on the isolation method, and all starches exhibited typical A-type X-ray patterns. The mean particle size of guinea starch ranged from 9.86 to 13.36 μm and showed round to polygonal shapes with pores. The isolation method impacted the glass transition temperatures and enthalpies, which varied in the range of 73.36 to 83.67 °C and 0.72-23.69 J/g, respectively. The pasting temperature also varied with the isolation method and followed the pattern of CA (84.80°C) > DW (81.95°C) > NaOH (79.82°C). All starches exhibited shear-thinning behaviour, and the storage modulus was higher than the loss modulus, indicating that the elastic component was higher than the viscous component. The NaOH-isolated starch has a high yield and amylose content and was chosen for the formation of nanoparticles. In the nanoprecipitation method, the particle size of starch nanoparticles ranged from 400 to 800 nm, and the zeta potential varied from -25.08 to -32.02 mV. The morphology of the SNPs showed both round and polygonal shapes, as well as some agglomerates. Native and octenyl succinic anhydride (OSA) modified starch nanoparticles were used to form an oil-in-water Pickering

emulsion. Microscopic images of 3% OSA-modified starch nanoparticles showed smaller and more stable droplet sizes for 35 days.



Dr. D.C. Saxena, Professor Dedicated to the continuing development and practice of creative teaching, innovative research and high impact public service programs that have improved food safety, food quality and processing. A **Seconded Faculty of Asian Institute of Technology, Bangkok (Thailand)**. Invited to many countries viz. Hungary, Germany, Thailand, Switzerland, U.K., Australia, Japan, Netherlands, Spain, UAE and USA to present my research work. Guided many students for their B.Tech.(27), M.Tech. projects (28) and Ph.D. thesis (18). Successfully completed the Leadership for Academicians Programme (**LEAP**) training of

MHRD at **IIT (BHU) India** (February 25th – March 9th, 2019) and (March 11th – March 15th, 2019) at **Penn State University in the United States**. Served as **Dean (Planning & Development) and Dean (Students Welfare) in SLIET**.

CONSULTANCY (04), PATENTS (02), PUBLICATIONS: Web of Science (SCI/SCIE) indexed (h-Index) 81 (25), Scopus Indexed (h-Index) 101 (28), Google Scholar (h-Index) 164 (33), AWARDS (06), RESEARCH PROJECTS (07), LIFE MEMBERSHIP in Scientific and Professional Organization/Societies (05), Books Authored (03), **RECOGNITION:**

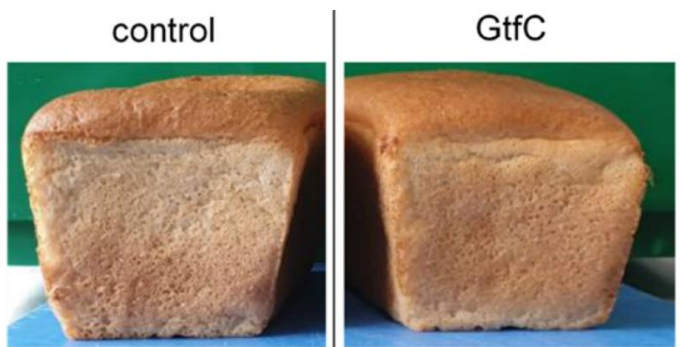
1. **Editorial Board Member of Nanoscience and Nanotechnology-Asia (2020)**
2. **Editor of Journal of Food Science and Technology (India) in 2012-2014**
3. Guided a **Post-Doctorate Fellow Dr. Mune Martin Alain, Lecturer, University of Maroua, Cameroon** under **Research Training Fellowship for Developing Country Scientists Fellowship**, Govt. of India. (July to December 2012)
4. **Member of Food Engineering Technical Advisory Group (TAG)**
5. **Expert Member of NBA Team** and in Panel of various Selection Committees of various Universities
6. **Member of Board of Studies** of various Institutes and Universities
7. **Organized 05 Conferences and 03 STTP**
8. **Guided 18 Ph.D. and 28 M.Tech. students** so far. 04 Ph.D. and 03 M.Tech. in progress.
9. Delivered various **Invited Talks** at **National** (CFTRI, Mysore, Imphal, CDLU, Sirsa, GJU, Hisar) and **International** (Germany - 04, Japan - 06, UAE - 01, Thailand - 01, United Kingdom - 01, Spain - 01)
10. **Presented** more than 97 papers in the 44 national and 50 international Conferences
11. **Published** about 15 Chapters in the books of renowned Publishers
12. **Two subject e-modules** have been developed under UGC, **e-PG Pathshala** viz. Technology of Meat, Poultry, Fish and Sea Foods and Technology of Spices & Condiments
13. Delivered various **Expert Lectures** for small-scale entrepreneurs under Entrepreneurship Development Programme organized by NITCON, at Barnala and Bhatinda in 2002 and 2005
14. **Developed a Training Centre** in Baking Technology at Badal Village, Punjab during 2002-03 for rural youths and women to train them for setting up their own bakery units.
15. **Research Associateship (CSIR) (1995)**
16. **Senior Research Fellowship (CSIR) (1990-1994)**
17. **Research Assistantship (DST) (1990)**
18. **GATE (1988)**

2.5. **Hans Leemhuis, Groningen (Netherlands)**

Changing the “starch” architecture using a-amylase family enzymes

Starch forms a major component of our diet, not only through the consumption of rice, corn, wheat, and potatoes, but also as a key ingredient of convenience foods. In these processed foodstuffs starch is being used to create a desirable texture at low cost. Most of the texturizing starches have seen some form of chemical, physical, or enzymatic modification to deliver the texture appreciated by customers. Because of the growing interest in simple and clean label food ingredients there is an increasing demand for functional starches manufactured without the use of chemistry. A second challenge is to develop functional starches that are slower digested, preventing blood glucose spikes. Is it possible to generate a series of functional starch derivatives ranging from fast, to slow, to slower digestion depending on the customer needs?

In this presentation the discovery and use of enzymes in the creation of functional starches will be discussed. The focus will be on the discovery and the use of enzymes with transferase activity on starch. As an example, I will present on the identification of a GH70 glucanotransferase of the microorganism *Geobacillus* sp. 12AMOR1 (GTFC), its biochemical properties, and how it changes starch. Subsequently I will present that this enzyme is capable of in situ starch modification in the process of bread baking. Importantly, gluten-free breads baked with GTFC maintained their softness relative to the control. During the presentation it will be discussed how GTFC changes the starch structure, and how this improves the bread texture.



Dr. Hans Leemhuis

Principal scientist at Royal Avebe, the Netherlands

I became fascinated in enzyme evolution when studying chemistry leading to a PhD in protein engineering. In the following years, I further explored how carbohydrate acting enzymes acquire their reaction specificity. I particularly focused on enzymes maintaining or generating polymeric carbohydrate products. Subsequently I became an industrial scientist, changing my focus towards the properties of starches made via enzymatic modification.

2.6. Guillermo Portillo, Aarhus (Denmark)

Environmentally friendly functionalization of starch via green deep eutectic solvents

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As one of the most abundant biopolymers in nature, starch has become the focus of study to produce bio-based materials, such as films and packaging. Nevertheless, further processing is often needed to obtain the desired mechanical and chemical properties of the target material. Often, these modifications require the use of harsh chemicals as solvents or catalysts, which can negate the green nature of starch-based materials. Therefore, a sustainable, catalyst-free, chemical modification of starch is proposed.

Previous work in our group showed that it was possible to obtain highly substituted starch using tartaric acid as organo-catalyst. This process also led to the crosslinking of starch branches through condensation reactions. Furthermore, the toxicity of semi- and nonvolatile migrated compounds in these materials was assessed, showing that, while some migration occurred, in-silico studies showed the compounds under analysis were of no concern regarding safety, according to European regulations.

As a following step, we worked on developing sustainable materials adhering as much as possible to the main principles of green chemistry, such as atom economy, less hazardous synthesis, and safer chemistry. A main focus of this research was exploiting the capability of natural deep eutectic solvents to work as reaction promoters and to solubilize starch. Among the screened mixtures, choline chloride/urea(ChCl:U) stood up as the best alternative, due to its low melting point, good starch solubility, and more basic pH.

An experimental design was developed around the catalytic system using ChCl:U as solvent/reaction promoter, starch, and acetic anhydride, with the goal of optimizing the reaction times and temperatures, while keeping high degree of substitution (DS). Esterification of the

material was confirmed by FTIR, ^1H and ^{13}C CP-NMR. Additional side product acetylurea was also formed under high temperatures. DS values of up to 3 were obtained at different conditions. Crystallinity (and lack thereof) of acetylate starch was determined by the deconvolution of the anomeric carbon (C1) peak in the CO-NMR analysis. Molecular weight was determined using HP-SEC MALS. While SEC-MALS analysis showed that the process caused limited chain degradation, SEM imaging showed that starch's granular structure was no longer present, instead replaced by slightly porous granules or strands. Thermogravimetric analysis showed the increase in thermal stability of the modified starches, as well as a higher glass transition temperature, with a much narrower transition range.

The results of the optimization highlight the efficiency of the process, where highly substituted material can be obtained in reaction times as short as 30 minutes, or temperatures as low as 110 °C, reducing the overall energy required. The use of $\text{ChCl}:\text{U}$ and starch, all from renewable sources and non-hazardous, improve the sustainability of the catalytic system.



Guillermo Portillo is a postdoctoral researcher at Aarhus University, working since 2022 in Dr. Mario Martinez's team. He uses his experience in the chemical industry, leading process optimization projects to develop novel, sustainable chemical synthesis to develop green materials. His research interests range from green chemistry and sustainability, to process simulation and mathematical modelling.

3. Application

3.1. Florian Wurm, Innsbruck (Austria)

The application of cationic starch as an auxiliary in reactive textile dyeing

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Reactive dyeing requires substantial amounts of salt in the dye bath to ensure effective fabric staining. Due to anionic dye molecules, as the anionic character of the fabric, electrostatic repulsion is partly overcome and impaired by salt addition. However, these salt loads burden the waste water stream, and several approaches have been investigated to eliminate the salt load. One possible approach is cationisation of the fabric. We followed this approach using cationised starch (CatS) as a dyeing aid. Starch properties can hereby be adjusted to process requirements. Besides, CatS biodegradability accords with cellulose fabric one's.

Cationic starch was deposited on cellulose fabrics via padding in a foulard. Additionally, selective citric acid cross-linking can prevent extensive CatS wash-off in the dyeing process. This enables starch fixation, and accordingly anionic dye affinity. Subsequently the CatS-coated fabric was immersed in the dye liquor for dye adsorption. Finally, sodium hydroxide addition enables covalent dye fixation on the fabric.

Performed experiments focused on dye uptake, as fabric properties of the CatS coated fabrics. Dye liquor concentration, and thereby fabric dye uptake, was screened over the course of the reactive dyeing procedure. Fabric stiffness, nitrogen content, colour and the dyeing result of the fabrics were evaluated. The dyeing protocol was adjusted over the process to enhance dye uptake.

Results suggest that CatS application can replace salt addition in reactive dyeing at a given level of fabric cationisation. Thus obtained fabric colours can be of similar intensity to common reactive dyeing protocols, if the dyeing process is optimised. In particular, longer dyeing times are beneficial. Conflicting, however expected, starch coating lead to fabric stiffening, an attribute that should be minimized or anticipated prior.



Dr. Msc. Florian Wurm, university education and work: 2006-2009 Bachelor of Science in physics, Leopold-Franzens-University Innsbruck, Innsbruck, BSc. in physics including various practica, 2009-2012 Master of Science in physics, Leopold-Franzens-University Innsbruck, Innsbruck, Function of an inlet system for acid-base measurements with a PTR-MS, 2013-2014 Phd program in physics, Universidade do São Paulo, Manaus and São Paulo (Brasil), Commencement as PhD-student at the University of São Paulo (SP) with regular trips to Manaus (AM), 2017-2020 Phd program in chemistry, Leopold-Franzens-University Innsbruck, Dornbirn (Austria), Implementation of Biopolymeric Gels for the Temporary Occlusion of Vessels in Medicine, 2020-present post-doc, Leopold-Franzens-University Innsbruck, Dornbirn (Austria), Implementation of Biopolymeric Gels for the Temporary Occlusion of Vessels in Medicine, non-university education and work: 2000-2003 Bank employee apprenticeship, Dresdner Bank AG, Rottach-Egern, 82/100, certified bank clerk followed by half a year working experience, 2005-2006 Work and Travel, Australia, 10 month trip with employments in different sectors, 2009-2011 Education as IFMGA mountain guide, BSPA Austria, Certified mountain- and skiing guide, 2010-2013 work, Mountain guide, Innsbruck, Guiding mainly in the southern and western alps, 2015-2016 work, Eventation GmbH, Innsbruck, Project manager for corporate events and meetings, semester abroad: 2008-2009 **University Bordeaux 1**, Bordeaux - Aquitaine, One semester of studies in France, measurement campaigns: CLOUD Measurements as part of the University of Innsbruck team, Collaboration from 16 different universities, participation in the 2011 summer campaign, GOAmazon Measurements as part of the University of São Paulo team, Collaboration from 11 universities and research institutions, participation in the buildup and two successive campaigns 2013 and 2014

4. Food / pharmaceutical applications

4.1. **Robert Gilbert**, Queensland (Australia)

Towards rice which is both healthier and tastier:

how endogenous proteins affect starch digestion in grain-based foods

Diabetes is becoming of epidemic proportions in many countries, including the US and China, where it has become a major and growing public health problem. In many Asian countries, it is likely that this is because of the large amount of rapidly-digested carbohydrates (rice and/or noodles) in normal diets. There is a strong need for developing food containing starch which is more slowly digested, and which also has appealing taste. Rice varieties have been developed that are slowly digested, and thus give slow blood-sugar release, which helps manage diabetes; however, they do not taste good.

We have explored the relations between starch molecular structure, rate of digestion and taste. The important structural feature is the chain-length distributions (CLDs) of amylopectin and amylose, for which we have developed mathematical models for the biosynthesis. These models enable the CLDs to be expressed in terms of a small number of biosynthesis-based parameters. The same has been done for in-vitro digestion rates; e.g. Li, Yu and Gilbert *Foods*, **11** 4012 (2022).

CLDs are composed of the contribution of various enzyme sets: one or more of each of a starch synthase, branching and debranching enzymes. The structural parameters are formulated by writing down the chemical-kinetic relations for CLDs for each set, in terms of the enzymatic activities controlling chain growth and stoppage, and solving these in the steady state, together with the restrictions needed for the chains to be able to crystallize. The resulting solutions enable the CLDs for each set to be expressed in terms of just two quantities in each enzyme set: the ratio of the activity of starch branching enzyme to that of starch synthase (β) and the relative activity of starch synthase (h).

CLDs are measured by first enzymatically debranching the starch, and then measuring the molecular weight distributions of the resulting linear chains by fluorophore-assisted carbohydrate electrophoresis (FACE; for shorter chains) and/or size-exclusion chromatography (SEC, also known as GPC, for chains of any length). The data are then deconvoluted into the contributions from each enzyme set, giving sets of β_i and h_i . Functional properties of interest include *in vitro* digestion rates, which can be fitted to simultaneous or sequential sets of first-order kinetics, which in turn can be reduced to a small number of rate coefficients. Given a sufficiently large data set, one can then carry out correlation analyses linking the sets of values of β and h for the structural

parameters, with functional properties such as digestion rate coefficients. As well as the parameters for starch, it is important to include proteins in this correlation analysis.

Another important quality is mouth-feel: in simple terms, whether or not the taste is acceptable. More sophisticated sensory analysis identifies more than 10 rice sensory qualities which are perceived by humans. Such analysis is expensive and complex, as it involves human panellists, and great care needs to be taken to avoid artifacts. However, once important sensory qualities have been established (for example, one of these for cooked rice is hardness), then correlations with instrumental sensory analyses can be carried out, and these can be used to validate – or disprove – these instrumental techniques, which are much quicker and cheaper, and thus can be used to amass statistically significant data. Again, correlation analysis with structural parameters can then be performed to find which structural features in the CLD, plus those in the proteins, are important for mouth-feel.

It is found that for rice and for noodles, the digestion rate is dominated by starch molecular structure, particularly the CLD, and that only certain CLD structural parameters (values of β and h for some enzyme sets) are important; the amount of protein is also significant. Fortunately, the enzyme sets of β and h which are important for rice mouth-feel are not the same as those which control rice digestibility. This has important conclusions for public health, especially in Asian countries, where rice and noodles dominate much of the food energy in normal diets, and where diabetes is a rapidly growing public health problem. At least for rice, where the complete sensory/digestion analysis has been performed, different components of the CLDs and proteins are found to dominate sensory and digestion. This has opened the way for plant breeders to develop rice varieties which have both acceptable taste and which also are slower to digest, by concentrating on genes which control separate aspects of the CLDs and of proteins. This has major implications for reducing the growing problems of diabetes.



Professor Robert G (Bob) Gilbert is Research Professor at both the Centre for Nutrition and Food Science, University of Queensland, and at YangZhou University, China, initially under the Chinese Government 1000-Talents Foreign Experts program. He spends half of each year in each institution. He received his undergraduate training at Sydney University, graduating in 1966, and his PhD from the Australian National University, graduating in 1970. He carried out postdoctoral work at MIT in the US from 1970 to 1972, and then returned to the University of Sydney, where he held a personal chair in polymer chemistry and was Director of the Key Centre for Polymer Colloids. In 2006, he took up a position in the Centre for Nutrition and Food Sciences at the University of Queensland to pursue his interests in the relations between human health and the structures of complex glucose polymers (starch and glycogen). He is a Fellow of the Australian Academy of Science (an Academician, equivalent to a Fellow of the NAS in the US), is author of about 530 papers, 6 patents, and 2 books (on unimolecular reactions and on emulsion polymerization). With 24,000 citations and an h-index of 77, he is one of Australia's most cited chemists. In reaction dynamics, he developed models and methods for predicting and fitting gas-phase rate coefficients of unimolecular and recombination reactions, and his methods are widely used for fitting and predicting data for combustion and atmospheric chemistry. In emulsion polymerization (the commonest means of making a wide variety of industrial polymers such as paints and adhesives, and a complex process involving many simultaneous steps), he developed a full understanding of all of the fundamental mechanisms, many of which were previously unsuspected or misunderstood. With these advances, it is now possible to polymerize simple systems and to predict the molecular architecture that will be formed under chosen conditions, while for more complex systems, trends can now be semiquantitatively predicted and understood. His mechanistic insights are used worldwide by industry. Over the last 20 years, he has extended this knowledge of synthetic polymers to the understanding and characterizing of branched natural polymers, particularly starch and glycogen. This led to unique combined experiment and theoretical methods for characterizing the complex molecular architecture of these biopolymers; the target is biosynthesis-structure-property relations important for human health (especially control and mitigation of obesity and diabetes) and industrial uses. He was President of the International Union of Pure and Applied Chemistry (IUPAC) Macromolecular Division (1998-2001), Elected Member of the IUPAC Bureau (2002-5), was Chair (1988-95) of the IUPAC Working Party on polymerization modelling and mechanisms, and was one of eight members of the IUPAC Strategy Development and Implementation Committee which carried out a major

reorganization of that body. He was Secretary of the International Polymer Colloids Group 1997-2001, and was Chair of both the Polymer and Physical Chemistry Divisions of the Royal Australian Chemical Institute. He is a winner of a Sydney University Excellence in Teaching Award, of the Smith Medal of the RACI in 1992 in recognition of outstanding research achievements in chemistry over the past decade, the Institute's Polymer Medal in 1995, shared the Australian Institute of Nuclear Science and Engineering Medal in 1993 for his work in understanding polymerization mechanisms, the RACI Olle Prize in 1996 for his book on emulsion polymerization, the RACI Physical Chemistry Medal in 1998, the RACI Applied Research Medal in 2005, the RACI Leighton Memorial Medal in 2007, the Ronald Ottewill Award of the UK Polymer Colloids Forum 2007, the Australian Academy of Science Craig Prize (2010), and the Paul J Flory Award in Polymer Characterization, Int. Conference on Polymer Characterization, 2017, and Jiangsu Province Science and Technology Cooperation Award, 2019. Editor of *Carbohydrate Polymers* (with the highest impact factor of all non-review polymer journals), 2016-20. His membership of editorial boards has included *Food Hydrocolloids* (current), *Carbohydrate Polymers*, *Biomacromolecules*, *Journal of Polymer Science and Polymer*, and he was Chair of the Gordon Research Conference on Polymer Colloids, 2003. He has chaired many conferences, including the IUPAC World Chemistry Congress and General Assembly (Brisbane, 2001) and IUPAC World Polymer Congress (Gold Coast, 1998), both of which had more than 1000 registrants. He is fluent in English (native speaker), French and German, and speaks basic Mandarin.

4.2. **Janusz Kapusniak**, Czestochowa (Poland)

Fiber and prebiotic substances from starch in human nutrition

Obesity and overweight, which are significant social problem, affect up to 20% of the developmental population and 50% of adults. The main factors contributing to the development of obesity include excessive consumption of products containing easily absorbable, high-calorie nutrients, including simple sugars. The development of obesity is also closely correlated with changes in the intestinal microbiota. Eating a diet high in dietary fibre and prebiotics is essential in the prevention and treatment of obesity and its complications. Oligosaccharides, mainly fructooligosaccharides (FOS), are commonly used as soluble dietary fibre with prebiotic properties. The main limitation of their use is that they cause gastric problems. Moreover, they are poorly tolerated by many people, especially with irritable bowel syndrome (IBS). The use of starch products, such as resistant dextrins, resistant maltodextrins and soluble corn fibre gives great opportunities in this regard.

The results of the research obtained as part of the project with the acronym PreSTFibre4kids are presented. This project is aimed at examining vegetable and fruit mousses with the addition of soluble dextrin fibre (SDexF) from potato starch with prebiotic properties, in terms of the prevention of overweight and obesity in children and the reduction of metabolic disorders secondary to obesity. In the first stage, an innovative method of obtaining SDexF on a semi-industrial scale was developed. Then, SDexF was subjected to comprehensive physico-chemical characterization and nutritional labeling. Based on analysis, including assessment of the composition and nutritional value, as well as safety assessment, National Institute of Public Health issued a positive recommendation recognizing SDexF as a food ingredient. In the next stage, industrial partner developed recipes of 6 flavors of vegetable and fruit mousses with and without addition of SDexF. The organoleptic characteristics of mousses were assessed using acceptance and preference methods according to the criteria developed in The Children's Memorial Health Institute. The most accepted and preferred: apple-carrot-quince, apple-peach-parsnip-lemon, apple-cherry-carrot-banana mousses were selected for further clinical trials. The study was performed in a group of 100 children aged 6 to 10 years, using a double-blind procedure. Evaluation points were anthropometric, metabolic, immunological parameters and changes in intestinal microbiota and metagenome.

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Janusz Kapusniak is a full professor of agricultural sciences in the field of food technology and nutrition employed at Jan Dlugosz University in Czestochowa (Poland). Since 2016, he has been the Vice-rector for Research and International Relations, and Head of Department of Dietetics and Food Studies. For several years he has been leading a group conducting research on physical, chemical and enzymatic modification of starch for food (dietary fibre, prebiotics, dietary supplements) and non-food (hydrophobically modified starches as packaging material, biomaterials of new generation from cereals and milling processing, microcapsules for controlled release). He is a vice-president of the European Polysaccharide Network of Excellence (EPNOE), a member of: the Committee on Food and Nutrition Sciences of the Polish Academy of Sciences, the Polish Society of Food Technologists - member of the Board of the Carbohydrate Technology Section, the Polish Chemical Society. Moreover, he is an expert of: Team of Experts of Conference of Rectors of Academic Schools in Poland (CRASP), the National Agency for Academic Exchange (NAWA). He has supervised 5 PhD students. Prof. Kapusniak published more than 100 peer-reviewed articles, 5 chapters in books. He is an author of 5 patents.

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

Organic pea starch – Optimization for the application in food matrices

Tim Terstegen, Eckhard Flöter, Marco Ulbrich

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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 available. Generally, pea starch has a higher amylose content and shows a variety of interesting product properties for example higher gel strength in comparison to other common starches.

A large variety of pea starches (smooth winter and summer pea varieties, grown in the same region and exposed to the same weather conditions and harvested in 2020 and 2021; n = 51) were analyzed. All varieties showed a similar starch granular size distribution and shape, and had almost the same amylose content. Surprisingly, basic functional properties which are essential for product characteristics differ extremely, depending on pea variety, growing period, and harvesting year. Even though, all investigated pea starches had a high amylose content above 30 %, some samples could not form a firm gel while others formed surprisingly strong gels. These extreme differences in functional properties also continued when comparing all varieties from the same cultivation period. Even when comparing mean values of each harvesting period in each year, gel strength varied resulting in a doubling in gel strength. Similar effects were observed when considering the hot paste viscosity. The pea starches analyzed spanned a broad field of different viscosity profiles. The variation in viscosity of the pea starches was larger than the difference of viscosity of starches from completely different botanical sources (e.g., potato and corn starch which were taken as reference).

However, also large differences regarding the molecular composition for the different pea starches were observed. While the difference of the weighted average molar mass (M_w) for the winter pea starches is mainly a function of the variety, the change of M_w for the summer starches depended more on the harvesting year and associated environmental condition (summer 2020 was extremely hot and dry). The M_w of the pea starches correlates with the viscosity profile. Samples with low M_w had in general a larger shear thinning behavior (high initial viscosity at low shear rates and considerably reduced viscosity at higher shear rates).

When considering the different M_w in combination with the results from the analysis of the pasting properties with differential scanning calorimetry a part of the variation in gels strength could be explained. Samples with lower M_w had stronger gels while higher M_w led to weaker gels. Samples which did not fit in this correlation were the same samples which had the highest respectively the lowest conclusion temperature during gelatinization. It is astonishing that native pea starches from the same cultivation region had such different functional properties.



Tim Terstegen, Ph.D candidate at the department of Food Process Engineering at Technische Universität Berlin, is part of a project which is focusing on the optimization of organic produced pea starches for usage in different food products. He published the first results of the project in a recent paper 'Organic pea starches - I. Comprehensive investigation of morphological and molecular properties'. He is a member of the Food Process Engineering Team since 2016 where he started as student assistant and had his first connection points to starch. He wrote his bachelor thesis focusing on the molecular investigation of starch gel structures whereas in his master thesis he focused on process technology and worked on fat fractionation using decanters.

4.4. **Jovin Hasjim**, Lestrem (France)

Roquette's journey in the development of glycemic carbohydrates with slow digestion

Roquette is a global leader in plant-based ingredients. One of our values is about well-being. Thus, many ingredients have been developed in Roquette to support healthy lifestyle, such as plant-based proteins, low-caloric sweeteners, and soluble dietary fiber with prebiotic effects. In the recent decade, Roquette has been developing glycemic carbohydrates with slow digestion properties. The targets of these ingredients are to prevent the postprandial high glycemic response (also known as hyperglycemia) and to provide a long-lasting energy source that is suitable for specialized nutrition, such as for diabetic patients and athletes.

Starch is the most abundant dietary energy source in the nature. It is synthesized in granular form by higher plants and can be found in grains, roots, tubers, stems, leaves, fruits, etc. The four main raw materials used by Roquette are corn, potato, wheat, and pea. Starch is composed mainly of two types of glucose homopolymers, which are amylose and amylopectin. Both molecules have α -1,4 and α -1,6 glycosidic linkages, but the ratios of these two types of linkages are different. The glucose monomers in the linear parts are joined together by α -1,4 glycosidic linkages, whereas the branching points are created by α -1,6 glycosidic linkages. Amylose is primarily linear with molecular weight ranging from 10^5 to 10^7 and about 3-5 branches per molecule, whereas amylopectin is highly branched molecules with molecular weight of 10^8 - 10^9 and about 5% branching points. The branches of amylopectin form double helices that create the semi-crystalline structure of the starch granules. Based on the amylose content, starch can be divided into waxy starch (0-8% amylose), normal starch (15-35% amylose) and high amylose (\geq 40% amylose).

Starch granules undergo a phase transition known as gelatinization when they are heated in excess water above certain temperature. Native starch granules are not soluble in cold water. During gelatinization, it loses the semi-crystalline and granular structure and turns into an amorphous paste. During this transition, the viscosity, the transparency, and the solubility of starch increase.

For nutritional purposes, starch is divided into 3 fractions: rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS). This concept was introduced by Englyst *et al.* [1]. They devised an *in vitro* digestion method to quantify these fractions. RDS is digested within 20 minutes, SDS is digested between 20 minutes and 2 hours, and RS is the remaining fraction not digested after 2 hours. Foods with high RDS rapidly increase the postprandial glycemic response causing hyperglycemia, which has been linked to metabolic diseases, including obesity and diabetes. Whereas, SDS and RS are deemed to be healthier as they maintain low postprandial glycemic response and thus can prevent metabolic diseases. In addition, SDS can provide a long sustained glucose supply as energy source for physical activities and brain functions, while RS can be fermented by the gut microbes to produce butyrate that can reduce the risks of colon cancer. In addition to the *in vitro* digestion method, glycemic index (GI) is also commonly used, which is a ranking system for carbohydrates based on their immediate effect on glycemic levels comparing with that of a glucose solution as the reference.

Native starch granules have been proposed to be good sources of SDS and RS [2], whereas gelatinized starches are known to contain high amounts of RDS as amorphous starch paste is more accessible to enzyme hydrolysis than the semi-crystalline starch granules. However, the amounts of SDS and RS in native starch granules depend on their botanical origins. Native starches with high amylose contents normally exhibit higher levels of SDS or RS, whereas native waxy and normal starches contain more RDS. Native pea starch granules, due to its higher amylose content than native waxy and normal maize starches, contain a lower amount of RDS, resulting in lower GI. Roquette has recently launched a granular pea starch with at least 30% SDS (Pea Starch LN30), which can be a good source of SDS or low-glycemic energy source for foods prepared with cold processes. In addition to Pea Starch LN30, native buckwheat starch exhibits a slower *in vitro* digestion rate when incorporated in biscuits than native wheat starch [3]. This is due to its high heat and shear resistance, allowing the starch granules to survive the baking process under low moisture condition, such as biscuit dough. Furthermore, the combination of starch and resistant dextrin (NUTRIOSE®) improves the texture of biscuits (comparing with only resistant dextrin) while providing a substantial amount of prebiotic dietary fibers and a low GI of 47 [4]. This is lower than the GI of regular digestive biscuits, which is between 55 and 62.

For specialized nutrition, there is a need for soluble carbohydrates with slow-release energy source. It is well known that increasing the degree of branching (or α -(1,6) glycosidic linkages) in starch molecules can reduce its digestion rate [5]. The α -amylases in the digestive tract cannot hydrolyze α -(1,6) glycosidic linkages, and thus having a higher amount of α -(1,6) glycosidic linkages can reduce the digestion rate of starch. A new development in Roquette is to branch starch hydrolysates (such as maltodextrin) using an enzymatic method, producing soluble carbohydrates with reduced digestion rate and essentially no dietary fibers, analyzed using the AOAC Method 2009.01 [6].

In conclusion, Roquette has recently gone through a new journey to develop glycemic carbohydrates with slow digestion, such as for specialized nutrition and healthy lifestyle. These carbohydrates provide energy slowly and gradually for an extended period. Native starches, such as pea starch and buckwheat starch, can be used to produce foods with slow starch digestibility and thus low GI values. However, there are some limitations in the applications of native starches, such as their low solubility and low heat resistance. To provide an alternative solution that is soluble and heat resistant, starch hydrolysates are branched using an enzymatic method to increase the ratio of α -(1,6) glycosidic linkages to α -(1,4) glycosidic linkages. The branched starch hydrolysates have slow digestion rate because α -amylases in the digestive tract cannot hydrolyze α -(1,6) glycosidic linkages. Roquette is still facing many challenges to develop glycemic carbohydrates with slow digestion. One of the main challenges is that the digestibility of starch can change after food processing due to the structural changes and the interaction with other components, and hence there is no one solution that can fit all applications. Roquette is determined to develop multiple solutions in order to cover a wider range of applications.

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Jovin HASJIM, Ph.D., *Ingénieur Recherche et Développement, Département Fonctionnalisation des Amidons, Sucres et Polyols, Roquette Frères, Lestrem, France*

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 and to create starch products with slow digestion properties. He has authored 12 patent applications, 41 peer-reviewed scientific articles, and one book chapter.

5. Future developments

5.1. Nelli Elizarov, Berlin (Germany) Sustainability in Transport through Renewable Fuels

In Germany in the transport sector, renewable fuels such as bioethanol, biodiesel or biogas have been the main contributors to climate protection and energy supply for several years. In 2022, the share of renewable energies in the transport sector was 6.8%, of which 90% were biofuels produced from cultivated biomass as well as from residues and waste materials.[1]

For the environment, climate protection and nature conservation, the raw materials used for the production of renewable fuels should be sustainable. Sustainability criteria are defined by legal requirements. The fulfilment of these criteria is reviewed and certified by recognized systems.

Legal basis

At the European level, the Renewable Energy Directive (RED II, (EU) 2018/2001) stipulates that in 2030 at least 14% of final energy consumption in the transport sector in each EU member state must come from renewable sources.[2] When using biofuels from cultivated biomass indirect land use changes (ILUC) must be avoided. This is achieved by allowing biofuels from cultivated biomass to count towards the transport target only up to a share of 7%. Biofuels with a high ILUC risk should no longer be eligible from 2030. To determine whether there is a high or low ILUC risk associated with a certain feedstock the EU has adopted a delegated act (EU) 2019/807.[3]

The RED II defines sustainability criteria for raw materials as well as criteria for GHG emissions from production facilities. According to these criteria, biofuels are only considered sustainable if they emit at least 50% less greenhouse gas emissions than fossil fuels - taking into account the entire production and supply chain. This percentage depends on the installation's operation starting date (Table 1).

Installation operation starting date	Transport biofuels	RFNBO*	Electricity, heating, cooling
Before October 2015	50%	--	--
After October 2015	60%	--	--
After January 2021	65%	70%	70%
After January 2026	65%	70%	80%

Table 1: Threshold for GHG emission savings for installations producing biofuels, bioliquids and biomass fuels in dependence of the operation starting date (REDII, (EU) 2018/2001 article 29). *RFNBO: Renewable Fuels of non-biological origin.

Furthermore, no land with high carbon stock or high biodiversity may be used to grow the plants for biofuel production. Likewise, only raw materials from sustainable cultivation may be used, for which detailed specifications are made from the point of view of nature conservation and environmental protection. Raw materials from primary forests, such as rainforest areas, are excluded.

In order to achieve the European targets, the German government has introduced a GHG reduction quota in the Federal Immission Control Act: Companies in the mineral oil industry are obliged to reduce the greenhouse gas emissions of the total volume of fossil fuels and biofuels placed on the market by a certain amount. Since the beginning of 2023, the obligation has been 8.0% and is set to rise to 9.25% from 2024.[4] To ensure the environmental compatibility of biofuels, the federal government has issued a Biofuel Sustainability Ordinance.

Certification of biofuels

Independent certification systems and bodies, such as REDCert and ISCC, which are recognised by the European Commission, are responsible for monitoring and controlling the entire cultivation, supply and production chain. [5,6]

In Germany, the Federal Agency for Agriculture and Food (BLE) approves and monitors the certification systems. In Nabisy, a web-based government database for the documentation and traceability of sustainable biomass supplies, the data on the sustainability of supplied biofuels is recorded by the economic operators (Figure 1).[7]

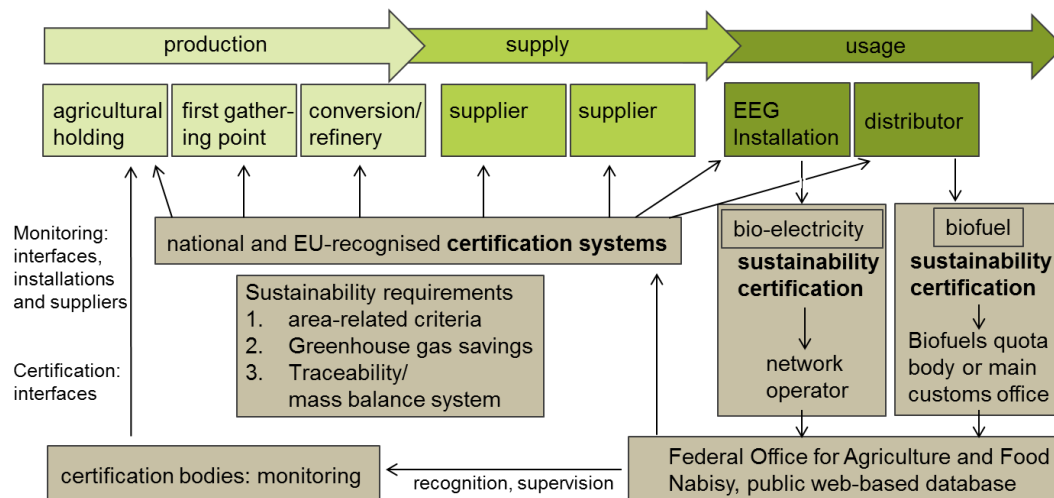


Figure 1: General procedure for sustainability certification by independent certification systems.

The total GHG emission saving of all biofuels was 84.5% compared to fossil fuels in 2021. By using biofuels instead of fossil fuels, around 11.2 million tonnes of CO₂ equivalent were avoided.[8]

- [1] Hintergrundpapier Erneuerbare Energien in Deutschland 2022, Arbeitsgruppe Erneuerbare Energien-Statistik (AGEE-Stat) am Umweltbundesamt, 2023
 [2] Renewable Energy Directive (EU) 2018/2001, <http://data.europa.eu/eli/dir/2018/2001/oj>
 [3] Delegated Regulation (EU) 2019/807, http://data.europa.eu/eli/reg_del/2019/807/oj
 [4] §37 BImSchG, <https://www.gesetze-im-internet.de/bimSchg/>
 [5] <https://www.redcert.org/>
 [6] <https://www.iscc-system.org/>
 [7] <https://nabisy.ble.de/app/start>
 [8] Evaluation- and Progress Report 2021, Federal Office for Agriculture and Food, 2023



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5.2. Arjen Postma, Haarlem (Netherlands)

New BioSolutions based on LpHera technology helps you to achieve more sustainable liquefaction, reduce energy and CO₂ footprint

Novozymes aims to build the future of sustainable business together with customers. Together with customers CO₂ emissions can be reduced in many segments by applying Novozymes biosolutions. These segments can for example be detergents, textile, vegetable oil or biofuel. The application of enzymes moreover enables less water usage and customers (producers) to produce more food with less raw material.

Novozymes moves together with customers towards a biorefining concept. The biological tools can be enzymes, microbes or yeast to convert many different feedstocks (e.g. corn, wheat, casava, soy, palm) into a wide variety of products such as renewable energy, food, feed, chemicals and materials.

All starch customers want to optimize the grain and starch processing process. Novozymes offers enzyme solutions in many steps of the process. This can be on the front-end application: in wet

separation process or downstream in starch refinery (liquefaction, saccharification, isomerization).

Recently, Novozymes has developed new products based on LpHera® technology which help to reach customers' sustainability goals. LpHera® was launched in 2014 and enabled customers to work at higher dry matter levels (DS) during liquefaction and generating more DX downstream after saccharification due to the generation of less panose precursors. Moreover, it can operate at pH 5-5.2 saving pH adjustment chemicals. This has many downstream advantages: less regeneration chemicals/ less wastewater, longer resin lifetimes and energy savings.

The recently developed new enzymes fit into the answers to customers' pains: increased Carbon Credit price, the overall emission allowance that will decline further each year towards 2050 and rising chemical prices.

Coming from the basis liquefaction solution Liquozyme® Supra, LpHera® has been the solution to go to higher DX levels and operate at lower pH. The recently developed **LpHera® Neo** and **LpHera® Duo** help in running the liquefaction at higher DS due to a good viscosity break and operating at even lower pH levels (ranging pH 4.3-5) and thus leading to further savings on water, chemicals and energy. The expansion of the toolbox in liquefaction solutions meets the variety of customer needs and helps in delivering CO₂ emission reduction.

Novozymes has developed an online tool for customers; the Refinery Optimizer that enables to calculate the savings of switching the alpha amylase to a newer generation product. Based on process parameters settings such as dosage, pH and the ion exchange parameters; a reference product (e.g Liquozyme® Supra) is compared to a new solution (e.g LpHera® Neo) and savings on chemicals, evaporation, regeneration and water are calculated.

Using this tool, it can be calculated that for a 1000 T DS/day starch producing plant, due to the risen chemical costs in one year, the value that LpHera® Neo can bring due to running the liquefaction at pH 4.8 instead of pH 5.6 (with Liquozyme® Supra) has increased by over 80%. Chemical prices and energy prices have risen substantially and therefore the savings on chemicals and evaporation due to making the switch from Liquozyme® Supra to LpHera® Neo have risen in this example from approximately 300.000 EUR/ year to 550.000 EUR/ year.

By combining the calculations from the Refinery Optimizer with further calculations and taking into account the natural gas price and CO₂ credit value, Novozymes has developed an indicative model to calculate overall savings. In this way, the value Novozymes liquefaction solutions can bring by operating at lower pH, higher DS and using less chemicals become clearly connected to the savings in tons of produced CO₂, and energy (gas and electricity).



***Arjen Postma** holds an MSc degree in Food Science and Technology from Wageningen University and has worked in his career with mainly (beet) sugar, inulin and starch in various roles in the industry. Since 2019 he is an Industry Technology Specialist for Novozymes providing technical application support for enzymes in the industry and is also involved in the development and roll out of new biosolutions.*

6. Technology

6.1. Vijay Singh, Urbana (USA)

Characterization and comparison of wet milling fractions of export commodity corn originating from different international geographical locations

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Soft endosperm corn has evident characteristics suitable for wet milling but is susceptible to breakage and fracture while transportation. In this study the millability of commodity corn samples with different endosperm hardness originating from different international locations has been

compared, and its economic impact on corn importers for wet milling has been discussed. The broken corn and foreign material (BCFM) for the soft endosperm US commodity corn ranged from 1.13 to 5.57% which was higher than other commodity corn from other international locations exported to the same country. US corn exported to different international markets showed higher starch yield in a range of 5 to 8% compared to the corn from different geographical locations exported to the same market. The excess starch from US corn directly translates to an additional revenue of 8.13 to 13 million USD/year for a 2540 MT/day wet mill plant. Therefore, it can be concluded that the soft endosperm US commodity corn, despite higher breakage, has superior millability and gives higher starch yields compared to the hard endosperm corn from other international locations which has comparatively lower BCFM. There are technologies that can be used to process broken corn separately after the initial cleaning process.



Dr. Vijay Singh is a Founder Professor at Grainger College of Engineering, Distinguished Professor of Bioprocessing in the college of ACES, Executive Director of Integrated Bioprocessing Research Laboratory at the University of Illinois at Urbana-Champaign and Deputy Director of the Center for Advanced Bioenergy and Bioproducts Innovation. His research is on the development of bioprocessing technologies for corn/biomass to ethanol, advanced biofuels, food and industrial products. Dr. Singh recent research activities are at the nexus of plant biotechnology and bioprocessing and are leading the development of new technologies and renewable products in the industrial biotech space. In his role at IBRL, Dr. Singh provides leadership in developing industrial partnerships, bioprocess pilot-scale proof-of-concept activities, and techno-economic analyses to facilitate commercialization of innovative technologies.

A fellow of the Cereal & Grains Association and the American Society of Agricultural and Biological Engineers, Dr. Singh has been a principal investigator or co-principal investigator for more than \$30 million research and \$202 million center grants, has authored more than 230 peer-reviewed journal articles. He was recognized with the 2003 Young Faculty Excellence Award, from Corn Refiners Association, the 2005 New Holland Young Researcher Award and the 2020 International Food Engineer Award from the American Society of Agricultural and Biological Engineers. The American Association of Cereal Chemists-International recognized him with Young Scientist Award (2007), Teaching Excellence Award (2013) and Applied Research Award (2018). Dr. Singh has also received College of ACES and College of Engineering Research Awards. In 2015, Dr. Singh was selected as a University Scholar, the highest honor given to a faculty member at the University of Illinois-system. Dr. Singh received his M.S. and Ph.D. in Food and Bioprocess Engineering from the University of Illinois at Urbana-Champaign.

6.2. **Ted Slaghek**, Wageningen (Netherlands) Electrification of the starch oxidation process

Roel Bisselink, Johan Timmermans, Michael Polhuis, Ted Slaghek

The oxidation of starch with sodium hypochlorite is a common practice in the starch industry. These modified starches are used both in food and non-food applications. Sodium hypochlorite is produced via electricity and subsequently transported to the starch modification factory. In this lecture we will present our research using the same electrochemical principles. Electrification of the starch oxidation process enables generation of hypochlorite by converting sodium chloride *in-situ* to hypochlorite, which could substantially lower the environmental impact, since brine waste streams are reduced, and increase safety as well, since transport, handling and storage of sodium hypochlorite is minimized. In the present study the influence of the electrochemical process on the quality of the oxidized starch is performed by evaluating the carboxyl content, molecular weight and pasting characteristics of oxidized potato starch. Secondly, the influence of re-using the spent oxidant on the quality of the oxidized starch is determined.



Ted Slaghek received his PhD in carbohydrate chemistry in 1989 at the University of Utrecht in the group of Prof Vliegthart on synthesis of capsular polysaccharide epitopes. He then moved to Japan for two year post-doc as a STA-Fellow at the RIKEN institute in the group of Tomoya Ogawa on the topic of synthesis of fragments of hyaluronic acid. He returned to Utrecht in 1992 as a Royal Dutch Academy of Arts and Sciences fellow continuing the research initiated in Japan. In 1993 he moved to Wageningen to the Agrotechnological Research Institute as head of the section carbohydrate modification. The focus of the research was the development of non-food applications for industrial carbohydrates. In 2001 he moved to TNO location Zeist and accepted the position of Product Manager Ingredients and Functionality. His research topics at TNO was development of new ingredients for food and non-food applications using natural polymers. In 2018 he moved back to Wageningen University and Research in the department of Wageningen Food and BioBased Research. He is currently a senior scientist with a research focus on the use of organic raw materials for on-food applications. He published more than 44 papers and over 45 patent applications in this research area.

6.3. **Maurice Essers**, Wageningen (Netherlands) Hydrophobic starches via reactive extrusion

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In the transition to a sustainable and circular society there is a need for biobased solutions to replace fossil derived products, especially in the food packaging industry where currently still synthetic (rigid) thin walled, water resistant containers are used in large quantities. These materials are not biodegradable, and the only solution is to collect and to recycle them. Thus, this industry is searching for biobased alternatives that enable to replace these materials. Native biopolymers, such as starch, can offer an interesting solution but most of them have hydrophilic properties, such as starch, which excludes them, at first sight, for these types of applications. To make them suitable for these types of applications, these biopolymers need to be modified in order to induce the desirable hydrophobic properties while maintaining their biodegradability. Various modification processes exist to induce water repellence into the starch chain via ester and etherification modifications, e.g., octenyl succinic anhydride modification of starch. These processes are traditionally conducted on granular starch under wet chemical conditions. In this project we explored this chemistry under conditions for reactive extrusion. Reactive extrusion has the advantage that modification and pre-gelatinization of the starch simultaneously takes place. This offers from a technological and commercial perspective interesting advantages. A precondition for reactive extrusion is that the modification must proceed within a short residence time. Our research pointed out that esterification of starch with octenyl succinic anhydride within the conditions of extrusion is successful and that different levels of substitutions can easily be achieved. The emerging starch derivatives were tested in in water soluble as well applications for water resistant containers. All hydrophobic modified starches turned out to have emulsification properties in solution, but their material properties were quite differently as expected. The hydrophobic substitution affected the porosity properties of the final material which promoted water absorption instead of inducing water resistance. This taught us that the modification on molecular level, affecting the physico-chemical characteristics of the biopolymer, will not consequently correspond to similar material properties and that this relationship is far more complex, especially in achieving hydrophobic properties. Hence, we started to develop new approaches in this field.



Maurice Essers, After attending the Joan of Arc lyceum in the Netherlands, I studied chemistry at the technical university in Aachen (RWTH) in Germany and graduated with the diploma thesis “Synthesis of UV curing polymers by living radical polymerization” under supervision of prof. dr. Hartwig Hoecker (DWI, Aachen). My professional working career as a chemist started at the R&D department of Sappi in Maastricht followed up in several global R&D departments of Cargill (Cedar rapids (USA)), Krefeld (Germany), Bergen op Zoom (the Netherlands) and Tate and Lyle (Aalst, Belgium). In 2009 I joined the carbohydrate research group of TNO in Zeist which merged with Wageningen Food and Biobased Research (WFBR) in 2018. Approximately 25 years of working history is built up in the field of carbohydrate processing and modification which also includes project management and acquisition of new projects. Currently, within WFBR, the focus of my carbohydrate research in collaboration with my colleague dr Lambertus van den Broek, is about clean label technology, bio catalysis and the development of sustainable solutions to utilize biomass for replacement of synthetic polymers. In this research both food and non-food applications are addressed. I am prime inventor of 10 granted patents for starch modification of which some have been implemented on commercial production level. Within Wageningen University and Research I am working in close collaboration with the research department Biobased Chemistry and Technology headed by professor dr Harry Bitter.

6.4. **Martin Heckl**, Freising (Germany)

Characterisation of starch granules with different analytical methods for use in printing materials for food 3D printing

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A new and innovative technology in food industries is the additive manufacturing, better known as 3D printing. By depositing the printing material layer by layer, it is possible to create defined and customized structures with new texture profiles. This simple process offers the opportunity to improve food sustainability by paving the way to on demand manufacturing. Developing new and innovative cereal-based bio-inks requires a deep understanding of the interaction between starch particles and added polymers which directly impacts the behaviour of the printing material during processing. Starch based bio-inks commonly contain proteins to enhance printability and the texture of the final product. Both polymers can interact through intermolecular forces. However, the impact of starch-protein-interactions on the printability of these materials, has not been elucidated yet. An analytical problem of particular interest is the concentration of proteins and lipids on the surface of starch granules due to possible interactions with protein polymers. Common methods to determine the protein and lipid concentration on the surface of starch particles are based on dissolution in different solvents. However, these strong chemicals might also affect the starch structure and residues may remain in the starch fractions. Therefore, a new analytical technique based on X-ray photoelectron spectroscopy (XPS) was developed to investigate the chemical composition of starch granule surfaces from different botanical origin (wheat, rice, potato). This surface-sensitive and quantitative technique uses X-rays to eject core level electrons of atoms at the surface of a solid (depth <5 nm). Based on the intensity of the photoemission line of a core level, the chemical composition of the surface can be deduced. In addition to that, we used Fourier transform infrared (FTIR) to further classify starch samples. This enables an unaltered characterisation of starch granules and, hence, sheds light on particle-polymer interactions in composed systems.

The main chemical components on the granule surface are carbon and oxygen, mainly related to carbohydrates but also lipids, and nitrogen, which is introduced by proteins. The surface analysis of different starches showed that the composition depends on the origin of the starch. The greatest amount of N was found at wheat starch surfaces, followed by rice and potato. Thus, wheat starch has the highest fraction of proteins on the surface and potato the smallest. Opposite to that, the C 1s/O 1s ratio is highest in case of potato starch, followed by wheat and rice. The amount on C on the surface of rice starch is the greatest but it has a lower C/O value. This means that the excess of carbon can be linked to lipids. Furthermore, the absence of phosphorus proves

the lipids to be mainly free fatty acids or triglycerides and not phospholipids. The results of the surface analysis demonstrate that XPS is capable of unravelling the chemical composition of different granule surfaces depending on the starch origin. Furthermore, the analysis of FTIR spectra reveal typical bands of starches from different origin. Using this knowledge, starch-polymer-interaction can be elucidated, and the functionality of composed systems can be explained in greater detail.



Martin Heckl studierte Lebensmitteltechnologie an der University of Applied Sciences Weihenstephan-Triesdorf (HSWT) und der Technischen Universität München (TUM). Vor seinem Studium schloss er eine Ausbildung als Konditor bei einer traditionellen Konditorei in München ab. Aktuell arbeitet er als wissenschaftlicher Mitarbeiter am Lehrstuhl für Brau- und Getränketechnologie der TUM an seiner Dissertation. Sein Fokus liegt auf dem 3D Druck von stärkebasierten Lebensmitteln.

6.5. **Lubbert Dijkhuizen**, Groningen (Netherlands)

Novel alpha-Glucanotransferase enzymes that introduce alpha1-6 or alpha1-3 linkages in starch, yielding soluble dietary fibers

The glycoside hydrolase (GH) family 70 originally was established for glucansucrase enzymes (e.g. GtfA of *Lactobacillus reuteri* 121) that convert sucrose into α -glucan polymers. We have identified enzymes constituting a first GH70 subfamily (e.g. GtfB of *L. reuteri* 121) inactive with sucrose, but displaying disproportionating activity with starch. GH70 members initially were found in lactic acid bacteria (<http://www.cazy.org/>). We have characterized a second GH70 subfamily (GtfC enzymes) in Gram-positive non-lactic acid bacteria, e.g. *Exiguobacterium sibiricum* 255-15 and *Geobacillus* 12AMOR1. GtfB and GtfC enzymes are biochemically related, catalyzing a 4,6- α -glucanotransferase reaction, cleaving (α 1 \rightarrow 4)-linkages and synthesizing consecutive α 1 \rightarrow 6 glucosidic linkages. GtfC enzymes have a non-permuted conserved domain organization, and are evolutionary intermediates between GH13 (mostly α -amylase and other starch modifying enzymes) and GH70 (glucansucrases/GtfB-like 4,6- α -glucanotransferases) family proteins. The products of these GtfB and GtfC enzymes are soluble dietary fibers. We have characterized a third GH70 subfamily (GtfD enzymes) e.g. in the Gram-negative bacterium *Azotobacter chroococcum* NCIMB 8003 and in the Gram-positive bacterium *Paenibacillus beijingensis* DSM 24997. GtfC and GtfD enzymes share the same domain architecture and display transglycosylase activity with starch and maltodextrins. However, these GtfD enzymes are unable to synthesize consecutive (α 1 \rightarrow 6) glucosidic bonds, instead they act on starch/maltodextrins to form higher molecular mass and relatively highly branched (>20%) α -glucans with (α 1 \rightarrow 4) and (α 1 \rightarrow 6) glucosidic linkages. These polymers are highly similar to the reuteran synthesized by the *L. reuteri* 121 GtfA glucansucrase from sucrose, regarded as a health promoting food ingredient. Most recently we have characterized a GtfB enzyme from *Lactobacillus fermentum* NCC 2970, displaying 4,3- α -glucanotransferase activity, converting amylose into an α -glucan with alternating (α 1 \rightarrow 3)/ α 1 \rightarrow 4 glucosidic linkages, and with (α 1 \rightarrow 3,4) branching points. This novel GH70 reaction specificity expands the range of α -glucans that can be synthesized from starch/maltodextrins, and allows the identification of key amino acid positions in the active site of the GtfB-like GH70 subfamily of enzymes, governing their linkage specificity.

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