

# Association of Cereal Research (AGF)

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In cooperation with

**Max Rubner-Institute**  
Institute of Safety and Quality of Cereal



## 64<sup>th</sup> Starch Convention

**April 24<sup>th</sup> – 25<sup>th</sup> 2013**

**in Detmold, Germany**

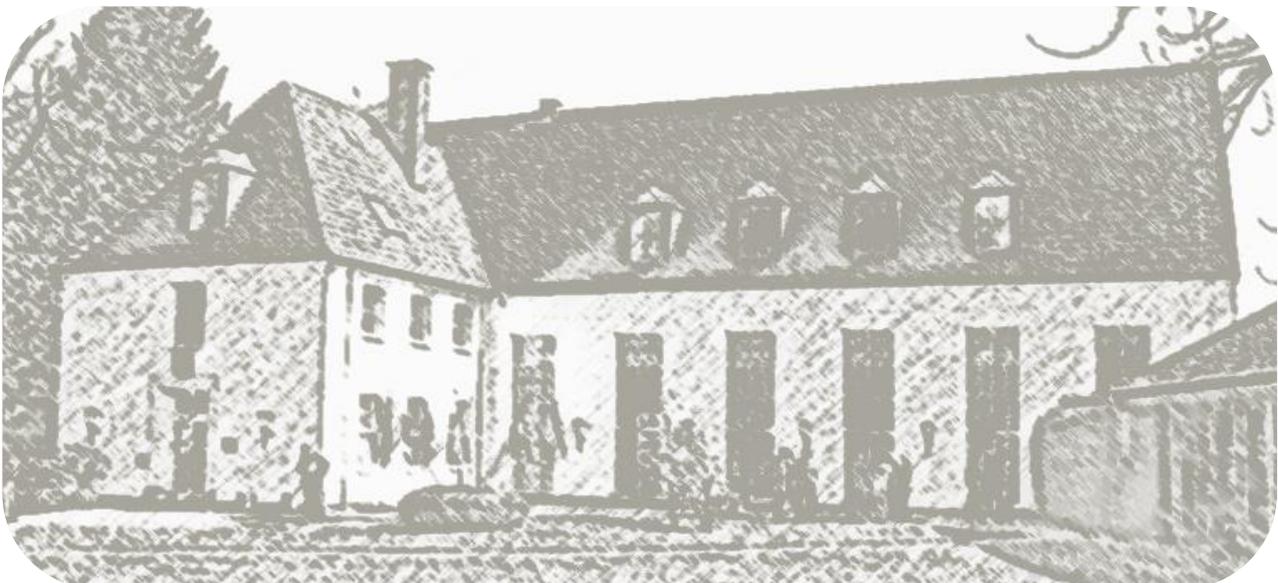
### Program

Evening Program

Exhibition

Participants

Summaries



## Wednesday, April 24<sup>th</sup> 2013

13<sup>30</sup> **Opening Remarks** by the President of the Association of Cereal Research,  
**Götz Kröner**, Ibbenbüren (Germany)

13<sup>45</sup> **Saare-Medal Awards Ceremony** for Dr. Bernd Kettlitz, Vilvoorde (Belgium)

### Session I

#### 1. Starch Structure and Characteristics (Session in parallel)

14<sup>15</sup> 1.1. **Eric Bertoft, Danusha Kalinga, Renuka Waduge** and  
**Koushik Seetharaman**, Guelph (Canada)  
Structure of starch in developing wheat endosperm

14<sup>45</sup> 1.2. **Scott Waitukaitis**, Chicago (United States)  
Impact-activated solidification of a dense cornstarch suspension

15<sup>15</sup> 1.3. **Jan A. Delcour**, Leuven (Belgium)  
Action patterns of maltogenic and maltotetraogenic alpha-amylases

15<sup>45</sup> 1.4. **Jasmien Waterschoot, Sara V. Gomand, Ellen Fierens,**  
**Jan A. Delcour**, Leuven (Belgium)  
Gelatinization properties of starch blends

### Session Ia

#### 2. Basics in Starch Technology - By Products (Session in parallel)

14<sup>15</sup> 2.1. **Berthold Wiege, Jürgen Hollmann,**  
**Heinz Themeier, Meinolf G. Lindhauer**, Detmold (Germany)  
Isolation of arabinoxylans from wheat bran by alkaline/hydrogenperoxide

14<sup>45</sup> 2.2. **Marnik Wastyn**, Tulln (Austria)  
By Products from Corn Starch Processing

15<sup>15</sup> 2.3. **Willi Witt**, Tecklenburg (Germany)  
By Products from Wheat Starch Processing

15<sup>45</sup> 2.4. **Do van der Krogt**, Veendam (The Netherlands)  
By Products from Potato Starch Processing

#### 16<sup>15</sup> Coffee Break

#### 3. Starch modification

16<sup>45</sup> 3.1. **Emma Qi, Richard Tester**, Glasgow (United Kingdom)  
Role of starch derivatives as pharmaceutical excipient

17<sup>15</sup> 3.2. **Inge-Willem Noordergraaf, Judy R. Witono, Leon P.B.M. Janssen,**  
**Hero J. Heeres**, Groningen (The Netherlands)  
Starch Modification through Graft Polymerization

#### 17<sup>45</sup> Exhibitor's Forum - short term presentations

# Evening Program

## Wednesday, April 24<sup>th</sup> 2013

After the Exhibitor's Forum "**Bread and Wine**"-Get-together in the "Haus des Brotes" (Exhibition Hall).

### Wine

#### Baden

2010er Winzergenossenschaft Bickensohl  
Grauburgunder, halbtrocken

#### Baden

2010er Oberbergener Baßgeige  
Müller-Thurgau, trocken

#### Baden

2010er Kirchberghof, Weingut Dr. Benz  
Spätburgunder Rotwein, trocken

#### Palatinate

2012er Dürkheimer Riesling  
Qualitätswein, trocken

#### Rheingau

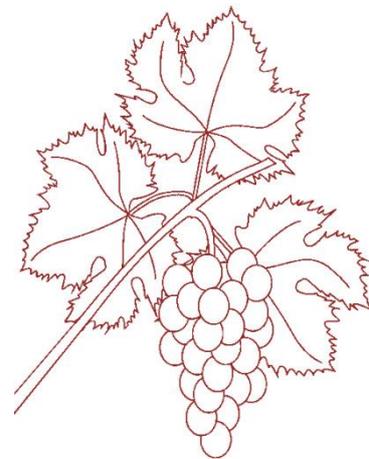
2011er Rheingau Riesling  
QbA, trocken oder feinherb

#### Wuerttemberg

2010er Schlossgut Hohenbeilstein  
Schillerwein, rosé, trocken

#### Wuerttemberg

2008er Winzergenossenschaft  
Trollinger, trocken



### Bread

Savoury snacks

Pretzels

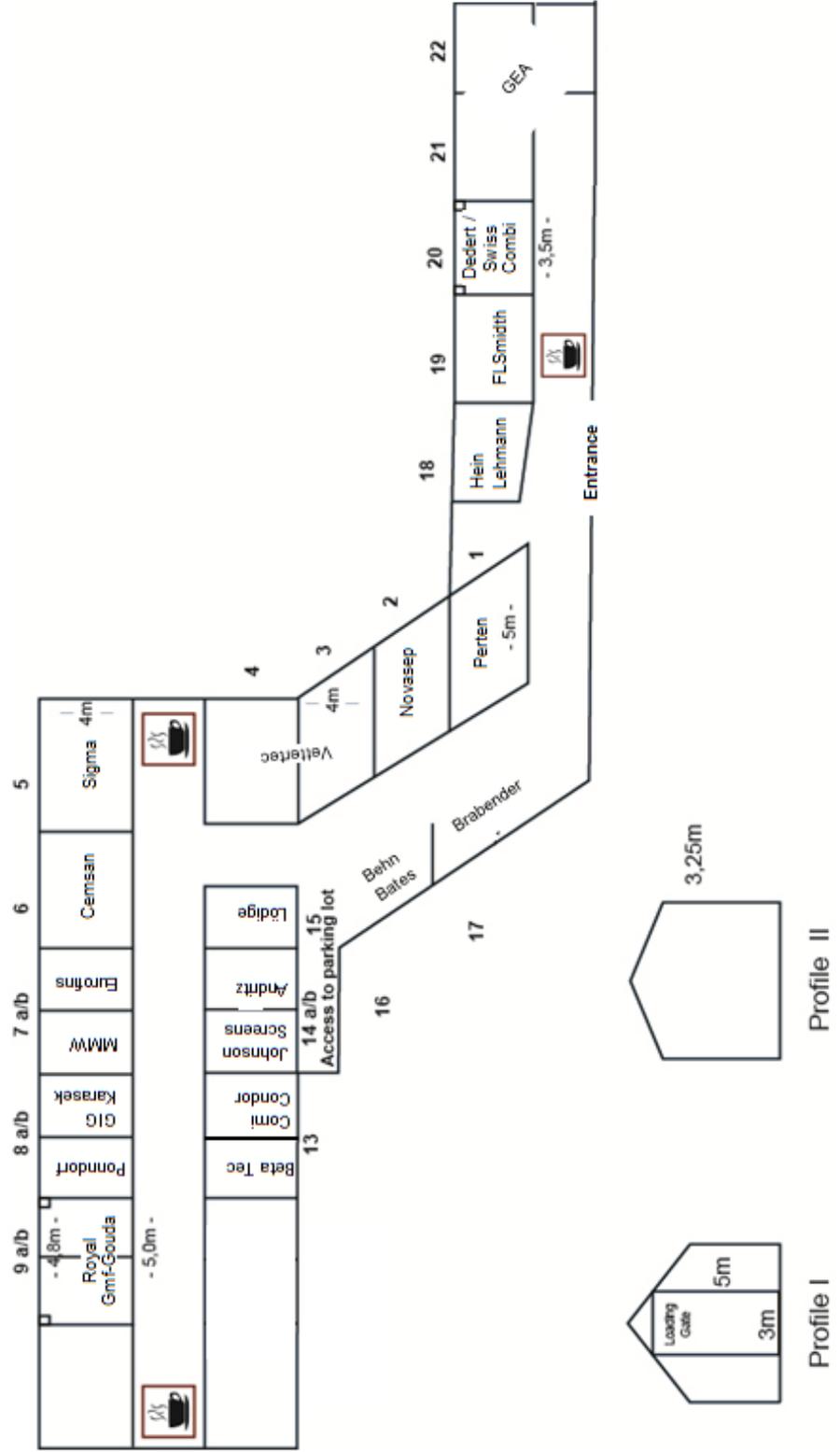
Baconsticks

20<sup>00</sup> **Social gathering** at Ristorante Italia, Krumme Straße 42, Detmold

**Thank you!**

# Exhibition Hall Association of Cereal Research Stand allocation

9th European Bioethanol Technology Meeting and 64th Starch Convention from April 23rd – 25th 2013



## Exhibition

**Andritz Feed & Biofuel Div.**, Cumming, GA 30040 (USA)  
**BetaTec Hopfenprodukte GmbH**, Schwabach (Germany)  
**Behn & Bates Maschinenfabrik GmbH & Co. KG**, Münster (Germany)  
**Brabender GmbH & Co .KG**, Duisburg (Germany)  
**Cemsan A.S.**, Arifiye, Sakarya (Turkey)  
**Comi Condor S.p.A.**, Santa Christina E Bissone (Italy)  
**Dedert Corporation**, Homewood (USA)  
**Eurofins Food Testing Netherlands B.V.**, Heerenveen (The Netherlands)  
**FLSmidth Wiesbaden GmbH**, Walluf (Germany)  
**GEA Westfalia Separator Group GmbH**, Oelde (Germany)  
**GIG Karasek GmbH**, Gloggnitz (Austria)  
**Hein, Lehmann Trenn- und Fördertechnik GmbH**, Krefeld (Germany)  
**Johnson Screens**, Availles-en-Châtellerault (Frankreich)  
**Lödige Maschinenbau GmbH**, Paderborn (Germany)  
**MMW Technologie GmbH**, Lutherstadt Wittenberg (Germany)  
**Novasep Process SAS**, Berlin (Germany)  
**Perten Instruments GmbH**, Hamburg (Germany)  
**Ponndorf Anlagenbau GmbH**, Kassel (Germany)  
**Royal Gmf-Gouda**, Waddinxveen (The Netherlands)  
**Sigma Process Technologies**, Atasehir/Istanbul (Turkey)  
**VetterTec GmbH**, Kassel (Germany)  
**Swiss Combi Technology GmbH**, Hendschicken (Switzerland)

## Exhibition

### **Exhibitor's Forum – Short Presentations (Wednesday, April 24<sup>th</sup>, approx. 6.00 p.m.)**

- 1. Dieter Abeln**, Behn + Bates GmbH & Co. KG, Münster  
Starch packaging into valve bags / High packaging outputs in small spaces with rotating packing systems
- 2. Kommer Brunt**, Eurofins Food Testing Netherlands BV, Heerenveen (Niederlande)  
Introduction of the Eurofins Carbohydrate Competence Centre
- 3. Tillmann Spies**, MMW Technologies GmbH, Lutherstadt Wittenberg  
Dry milling of wheat for starch and bio products
- 4. Mesut Kemal**, CEMSAN A.S., Arifiye/Sakarya (Turkey)  
Company Presentation

# Participants

Effective April 18<sup>h</sup> 2013

Abel, Christian	GEA Wiegand GmbH, Ettlingen
Acildi, Emre	CEMSAN A.S., Arifiye/Sakarya (Turkey)
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Agha, Diana	Pharmazeutische Technologie Rheinische Friedrich-Wilhelms-Universität Bonn
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## 1. Starch Structure and Characteristics

- 1.1. **Eric Bertoft, Danusha Kalinga, Renuka Waduge and Koushik Seetharaman**, Guelph (Canada)  
Structure of starch in developing wheat endosperm

The development of starch in wheat kernels was followed through pre-physiological maturity to post-physiological maturity. At 3 days after anthesis (DAA) the kernel contained only pericarp tissue, in which small starch granules of transitory type had already developed. At 7 DAA endosperm was developing and contained small, lenticular shaped starch granules, which later apparently became the population of large granules. Small granules had started to develop in the endosperm at 14 DAA and at 21 DAA the pericarp had disappeared. Starch granules from 7 DAA and 14 DAA, which were mixtures of endosperm and pericarp starch, already possessed an ordered crystalline structure and blocklets with a fuzzy contour were observed by AFM on the surface of the granules. Later, the blocklets obtained a more defined contour. Apparent amylose content was initially low; at 3 and 7 DAA the structure of the apparent amylose suggested that it in fact consisted of a material of intermediate type between amylose and amylopectin. The fine structure of the amylopectin component was very similar throughout the development with small, but distinct, differences between the large and small granule populations. However, the clusters became slightly larger at the end of the pre-maturity stage (28 DAA). It appeared that the external structure of the amylopectin was less organised at this stage. During post-physiological maturity, when no net synthesis of starch occurs and the kernel starts to dry out, still some changes in the amylopectin structure were noted, possibly due to trimming of the external chains by branching and/or debranching enzymes. This apparently resulted in a slight decrease of the size of the clusters.

- 1.2. **Scott Waitukaitis**, Chicago (United States)  
Impact-activated solidification of a dense cornstarch suspension

A dense suspension of cornstarch and water exhibits unique behaviors that fascinate scientists and children alike. When poked lightly the suspension flows like a fluid, but when punched or kicked it seems to miraculously "solidify" and provides enormous resistance to the motion of the impacting object. This allows, for example, a full-grown person to run across the suspension surface, giving the crowd-pleasing impression that humans can "walk on water." Using an array of experimental techniques, including high-speed video, embedded force and acceleration sensing, and x-ray imaging, we investigate the dynamic details the impact process as it unfolds. We find that the impacting object drives the fast growth of a jammed region of cornstarch grains directly below the impact site. Being coupled to the surrounding fluid by grain-mediated lubrication forces, this creates substantial peripheral flow and ultimately leads to the rapid extraction of the impactor's momentum. With a simple jamming picture to describe the solidification and an added mass model to explain the force on the rod, we are able to predict the forces on the impactor quantitatively. These results not only explain a crowd-pleasing trick, but also shed light on the inherently non-equilibrium nature of flows in dense suspensions.

1.3. **Jan A. Delcour**, Leuven (Belgium)  
Action patterns of maltogenic and maltotetraogenic alpha-amylases

α-Amylases are increasingly used in several industrial processes, such as starch liquefaction, detergent systems and breadmaking. The susceptibility of starch to amylases not only depends on the properties of the substrate, but also on the characteristics of the amylase. A maltogenic α-amylase from *Bacillus stearothermophilus* (BStA) has interesting properties for use in industry. This amylase with high temperature stability specifically releases maltose by shortening the outer chains of starch polymers. These properties are important when considering their use in maltose syrup production and as anti-firming enzyme in breadmaking. Another industrially used amylase is a modified malto-tetraogenic α-amylase from *Pseudomonas saccharophila* (PSA) with high temperature stability. This amylase preferentially releases maltotetraose from the outer chains of starch polymers. However, the precise action pattern and/or specificities of these enzymes are not yet known. Indeed, the action pattern of PSA has, to the best of our knowledge, not been described. In this study, a thorough comparison was made between the action patterns of both BStA and PSA. Hereto, the action pattern was studied on amylose, amylopectin as well as on α-limit dextrins. The impacts of PSA on these polymers differ from those of BStA. BStA has a higher ratio of exo- to endo-attack than PSA, thereby altering the starch polymer structure less than PSA does. Since these enzymes are frequently used in industrial processes with increased temperature, the effect of increased temperature on the action pattern was also studied. While both enzymes were optimally active at about 60 °C, BStA at higher temperatures displayed a relatively higher endo-activity than at lower temperature, while this was not observed in the case of PSA.

1.4. **Jasmien Waterschoot, Sara V. Gomand, Ellen Fierens, Jan A. Delcour**,  
Leuven (Belgium)  
Gelatinization properties of starch blends

In some food applications, chemically modified starches outperform their native starch counterparts. Because of the growing consumer interest in clean label starches, it is relevant to study the functionality of blends of native starches. In this study, gelatinization properties of starch blends were investigated. Potato, maize, waxy maize, rice and waxy rice starches were blended in different ratios and the influence of granule size and amylose content on the gelatinization properties of blends in excess water [1:3 starch dry matter (dm):water] and at intermediate water content (1:1 starch dm:water) were studied. Interactions between the starches took place in excess and intermediate water contents, the effects being larger at intermediate water content. Competition for water delayed the gelatinization of potato starch in the presence of waxy rice or maize starch. The start of gelatinization of the other starch in the blend was in each case shifted to a lower temperature. We speculate that this is due to the difference in rate of water absorption between the granules due to different ratios of surface area to volume, resulting in an unequal distribution of the available water. Blends of starches with the same granule size (rice – waxy rice or maize – waxy maize) showed less competition for water.

## 2. Basics in Starch Technology - By Products

2.1. **Berthold Wiege, Jürgen Hollmann, Heinz Themeier, Meinolf G. Lindhauer**,  
Detmold (Germany), **Sylvia Radosta, Waltraud Vorweg**, Golm (Germany)  
Isolation of arabinoxylans from wheat bran by alkaline/hydrogenperoxide

Wheat bran is an important by-product of the wheat milling industry and accounts for 11 to 19% of the grain. It mainly consists of dietary fibre (43-53%). The fibre fraction

includes arabinoxylans (AX) (38-55%), cellulose (16-30%), lignin (5-20%) and other nonstarch polysaccharides (Rose and Inglett, 2010; Maes and Delcour, 2001; Hollmann and Lindhauer, 2004). Depending on their molecular masses, AX may have different health benefits in the large intestine (Gemen, de Vries and Slavin, 2011). Process simulations indicated that an AX product of 80% purity could be produced at costs of around 3.7-4.5 £/kg (Misailidis et al, 2009).

The aim of this study was to develop a small technical process in a scale up to 150 litres of slurry and to determine important parameters of the process steps necessary for developing a process for an economic production of AX. Experiments were carried out in a laboratory and in a small technical scale up to 10 kg of wheat bran in one batch. Initially the starch was mechanically separated at room temperature by aqueous dispersion and wet sieving. Then alternatively three different processes were carried out by always using this destarched wheat bran.

First, the AX were extracted from the wheat bran (3.46 kg before destarching) in a small technical scale by alkaline/hydrogen peroxide at 60°C followed by centrifugation, ultra- and diafiltration, ethanolic precipitation of AX and drying. The purity was 69.8% AX in d.m. and the xylose/arabinose-ratio was 2.38.

Second, the wheat bran was extracted with water only in a laboratory scale (400 ml autoclave) at temperatures in the range of 147-163°C with extraction times of 0.5-2.0 h. The resulting suspension of the autoclave-extraction was centrifuged and the AX were isolated from the supernatant by ethanolic precipitation using a mass ratio of ethanol (96%) to supernatant of 3.17 to 1. The precipitated AX were then centrifuged and freeze dried. The AX content was 47.3-58.6% in d.m..

Third, the wheat bran (10.00 kg before destarching) was extracted in a 150 litre pressure-reactor with water only at 147-163°C for 0.5-1.0 h. The dispersions were sieved (250µ), the sieve residue washed, the liquid phase then alkalinely (pH=10.0) bleached with H<sub>2</sub>O<sub>2</sub> at 23-49°C and concentrated and purified by membrane filtration using polysulfone membranes with a molecular cut off of 10.000 g/mol in a 1.05 sqm plate and frame process unit at 55°C. Starting with approx. 160 kg, a concentration up to 45 kg was possible. The resulting AX concentrate suspension was then spray dried at 190/90°C. By this process the lowest AX content (37.6-45.1% in d.m.) in the final product was obtained.

The molecular masses of the AX isolated in the laboratory scale strongly depended on the extraction conditions and varied in the range of Mw = 11.100 to 220.000 g/mol. With increasing extraction time and temperature the polymeric carbohydrates were distinctly degraded. The molar masses Mw of AX extracted under alkaline/hydrogen peroxide conditions were about 70.200 g/mol.

Keywords: Arabinoxylan, wheat bran, isolation, process parameter

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## 2.2. **Marnik Wastyn**, Tulln (Austria) By Products from Corn Starch Processing

Global starch production was 35 million tons in 2011, with corn starch making up the largest share (43%). In Europe in the same year, annual corn starch production amounted to 4.7 million tons. The raw material corn (*Zea mays* L.), a type of grain belonging to the grass family, is made up of around 70% starch, but also contains proteins, fat and fibre, which are extracted as economically valuable by-products of industrial processing. The amount of by-products resulting from starch production each year corresponds to some 2.7 million tons/year worldwide and 0.8 million tons/year in Europe.

The lecture will outline the industrial process for the extraction of corn starch and the resulting by-products corn germ, corn steep liquor, corn gluten meal and corn gluten feed. The composition and properties of these valuable by-products, as well as their uses as animal feed will be described in detail. Lastly, specific aspects, such as mycotoxin contamination, non-feed applications and potential uses for by-products from corn starch production will be discussed.

## 2.3. **Willi Witt**, Tecklenburg (Germany) By Products from Wheat Starch Processing

Wheat starch is one of the important starch resource which is manufactured worldwide, but especially in Western Europe.

A certain specialty is that the starch out of the wheat consists of two fractions. These fractions are the A-starch granules and the B-starch granules. Wheat flour contains round about 70 % of recoverable A-starch with an amount of approx. 70 % A-starch granules with a diameter bigger than 10 microns and 30 % of B-starch granules smaller than 10 microns. Another fraction is the so-called C-starch. This starch fraction doesn't have any visible starch granules under the microscope. The analytical starch consists of starch polysaccharides like hemicellulose, fibers, pentosanes and others. The wheat and flour processing in a dry and wet process is resulting in bran by product from the flour mill and the A-starch or primary starch as main product from the wet process. The main by product is the wheat gluten, a protein containing fraction from the wheat. The second by product is the B-starch or secondary starch. The pentosanes and soluble of the wheat flour can be treated in a thermal and enzymatic process step prior to evaporation. The concentrate of the evaporator is then preferable be dried in combination with the brans from the flour mill to a wheat gluten feed product, which is mainly used for animal feed.

The recovery of the starch granules is not easy due to the presence of the high amount of the B-starch granules. On the other hand the viscosity during the operation is limiting the complete extraction of 100 % of the starch granules as final A-starch product. That is requiring enough clarification area and G-force to ensure the highest as possible A-starch yield. Separators are used for the pre concentration of the starch milk and recovery of small granule starches as well the production of process water, which is reused in the different process steps. Hydro-cyclones mainly used as washing device for the A-starch to remove the soluble proteins, minerals and no polysaccharides and insoluble fine particles.

Nearly 50 % by weight and less than 50 % by value are by products. These by products are used as food, feed as well technical product. A clear definition about by-products is not really given. By-products are very important for the economy of a starch factory.

## 2.4. **Do van der Krogt**, Veendam (The Netherlands) By Products from Potato Starch Processing

Since starch is the main product from processing potatoes to starch, in principle all the remaining components are by products. Some of these components can be manufactured as pure chemicals, but most of them are manufactured as composed streams. In the early 20th century potato starch factories were rather small and had large water consumption per ton of processed potatoes. There was only one by product: the potato fibres. The diluted potato juice was simply spilled into the canals. Consequence of this practice was pollution of the surface water, foam building on surface water and a terrible smell during the potato campaigns. It took the industry over 75 years to find a solution for this spilling. From approximately 1970 protein factories, evaporation installation and aerobic waste water treatments were built at all AVEBE factories. The basis for this development was found in new starch processes with low water consumption (factor 20, compared to the old processes).

Since by products are coming with the potatoes, process solutions for by products must be very robust and handle the whole stream of by products. To have a process that treats for example only 10% or even up to 75% of the total stream is not a feasible option. It is however possible to implement processes on smaller scale to produce high end products parallel to the total solution. The existing potato process, including producing by products, is a high energy consuming process.

Future developments should be focused on processes with lower energy, water and chemical consumption. One of these developments is Solanic. This company focuses on producing functional potato protein for the food market, by using new and innovative processes based on low water, energy and chemical consumption.

Another development is the production of biogas in an anaerobic waste water treatment. The challenge of such a biogas installation is to find a feasible way of handling the effluent in sustainable way. Production of algae can be one of the options for the future.

## 3. Starch modification

### 3.1. **Emma Qi, Richard Tester**, Glasgow (United Kingdom) Role of starch derivatives as pharmaceutical excipient

Excipients provide many functions in pharmaceutical systems although they are not the active therapeutic agent per se. They make possible dosage formats by providing carrier, structural and functional aspects to the formulation. The use of excipients in pharmaceutical systems is both a science and an art. Many factors need to be considered when utilising excipients and the range of materials available for the purpose can make this quite a difficult decision. The production hurdles associated with making the formulation and end point functionality drives the choice of material. With starch based excipients there are many products of choice – native starches (from a range of botanical sources with associated size, architectural and compositional variation), physically modified starches (pre-gelatinised especially), enzymatically modified and chemically modified. Whilst these molecules are  $\alpha$ -glucans, their functional (and physiological role and impact) can be very different. Further processing steps (e.g. agglomeration) provide other functional opportunities. Cost and legislative control of excipient usage must be considered in pharmaceutical formulations. In addition, a knowledge of how the active pharmaceutical ingredient (API) interacts with the excipient is very important in terms of dosage and bioavailability. One

big advantage of starches and starch derivatives (within reason) are generally regarded as safe (GRAS) providing confidence for usage in a range of formulations. The presentation seeks to highlight some of the functional variations of  $\alpha$ -glucans and their associated benefits in certain formulations. Specific examples of functionality are drawn from the literature together with work of the authors.

3.2. **Inge-Willem Noordergraaf, Judy R. Witono, Leon P.B.M. Janssen, Hero J. Heeres**, Groningen (The Netherlands)  
Starch Modification through Graft Polymerization

### Introduction

An important and actual topic in chemistry and chemical engineering research is the substitution of traditional feedstocks, fossil fuels derived chemicals, by feed materials and products based on biopolymers. Graft copolymerization of starch offers the prospect to replace many of the currently applied synthetic polymers in industrial and consumer products. In a joint research project at Groningen University and UNPAR at Bandung, Indonesia, graft polymerization reactions of cassava starch with acrylic acid have been investigated. There are many potential applications of starch with hydrophilic grafts, like water absorbing materials, thickening agents, detergent co-builders and flocculants.

### The grafting reaction system

The grafting reactions were carried out with gelatinized cassava starch and Fenton's reagent was applied as an initiator. Figure 1 shows the principal reactions. Free radicals are created that can activate starch sites, to react with monomer molecules into a grafted copolymer side chain. However, in the homogeneous reaction there is also a side reaction: the formation of the homopolymer of acrylic acid.

### Results and discussion

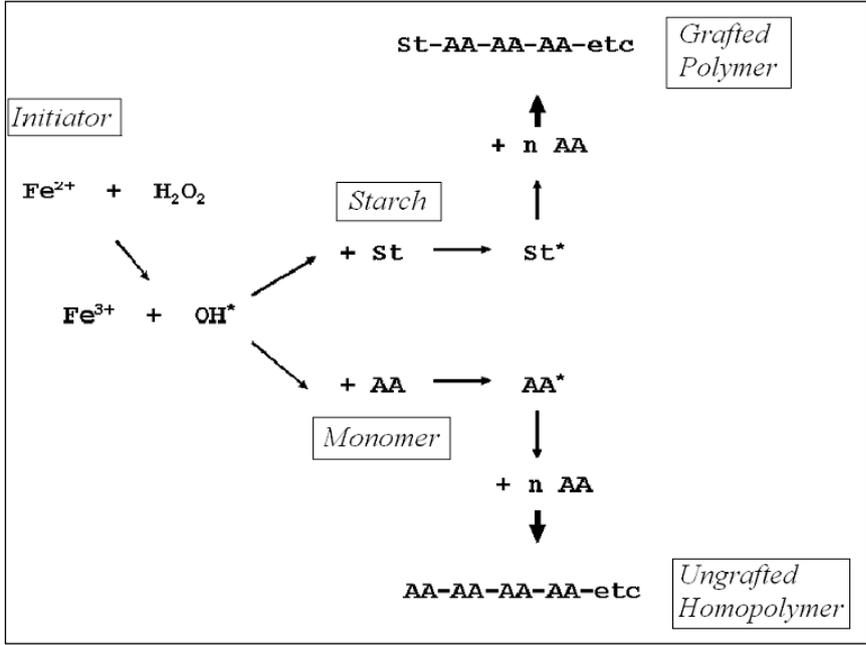
An experimental design method was applied to assess the importance of the many reaction variables as well as to search for optimal conditions. The formation of homopolymer appears to be inevitable, as it was found that grafting efficiency is in the order of 30-40%. The amount of grafted side chains in the product is ca 15 wt% at those conditions.

When homopolymer formation can not be avoided, perhaps the primary applications of the product must be found in systems where the presence of homopolymer can be tolerated. Thickening agents is such an application. We found that the thixotropic properties of a starch gel can be reduced or almost eliminated by increasing the ratio of monomer to starch in the reaction system. This is illustrated by the torque curves shown in Fig.2. Another interesting application of starch grafted with acrylic acid is in materials that can absorb large quantities of water, so-called superabsorbents. A polymerization crosslinker is applied to form the gel network that absorbs the water. It was found that a substantial portion of the homopolymer is taken up by the crosslinker into the final network. Fig.3 shows the water absorption capacity (WAC) of the grafted starch at various levels of crosslinker dosage. Interestingly, the optimum product could retain up to 63 g H<sub>2</sub>O/g under severe suction, showing that this material combines a good absorption capacity with sufficient gel strength.

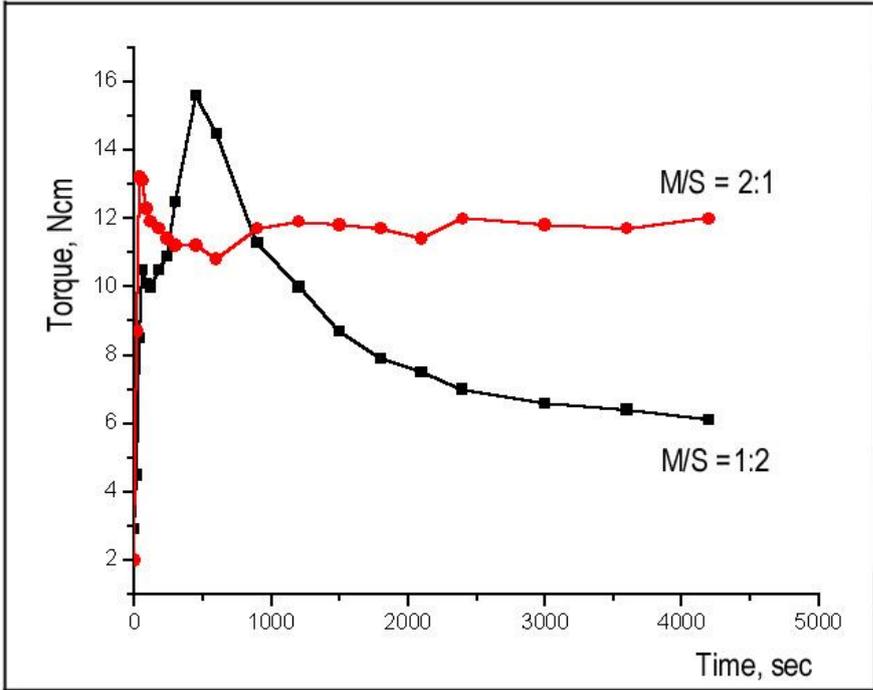
The results summarized here were reported in the PhD Thesis of Mrs. J.R. Witono (Groningen, September 21, 2012). A follow-up project is currently being started at UNPAR, Indonesia, to further explore the potential of water absorbing grafted starch in agricultural applications.

### Figures

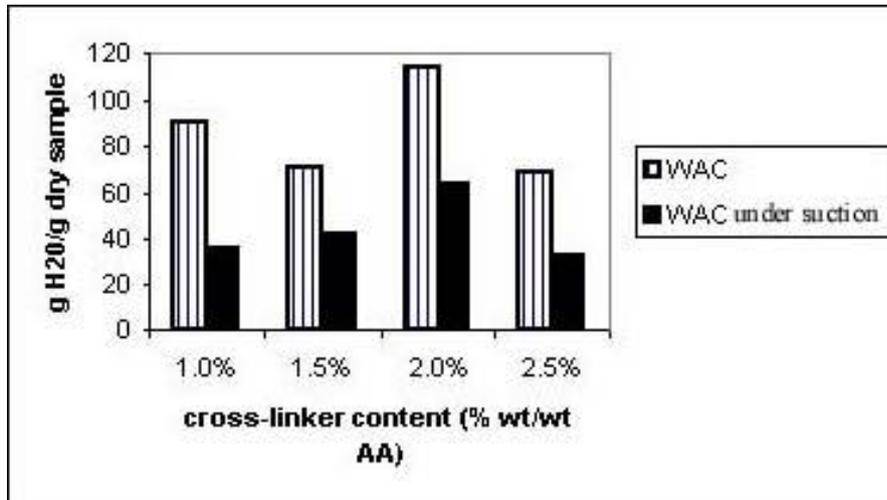
**Figure 1:** Reaction scheme of the graft copolymerization of starch with acrylic acid.



**Figure 2:** Torque development for grafted cassava starch at two ratios of Monomer-to-Starch.



**Figure 3.** Water absorption capacity and retention under suction, at various levels of crosslinker dosage.



## 4. Starch Market

### 4.1. Harald von Witzke, Berlin (Germany)

The End of the Agricultural Treadmill: Prospects for EU Agriculture

The worldwide request of agricultural products will increase more than 100% in the first half of the 21st century. This fast request can be satisfied by an expansion of agricultural areas or by increasing the efficiency of the already used areas. The first option is only available to a limited extent. On the other hand it will be difficult just to increase the efficiency to the needed amount. In consequence of this the tendency of the agricultural prices will point upwards.

Within the lecture we will analyze the reasons and will see the consequences for agriculture, world food, climate protections and biodiversity

## 5. Starch Analysis

### 5.1. Carolin Menzel, Uppsala (Schweden)

Analysis of citric acid cross-linked starch films

The effect of citric acid (CA) on starch films has been examined. A new method to detect cross-linking of starch by CA in solution-cast films by molecular weight measurements is described. Furthermore, we managed to distinguished between free, mono- and di-esterified CA and quantify di-ester content within starch films by using a modification in the method of complexometric titration with copper(II)-sulfate. Cross-linking of starch by CA occurred at low temperature, 70 °C, which we assumed is so far the lowest temperature reported where cross-linking reaction occurred. This is essential for starch coating applications within paper industry since no high temperatures for curing will be required. However, curing at 150 °C and high CA concentrations, 30 pph, increased cross-linking reaction. Furthermore, the physical properties like water solubility, gel content and glass transition temperature, were highly reflected by the molecular structure i.e. cross-linking and hydrolysis, as well as plasticizer content and curing temperature.

## INTRODUCTION

In recent packaging research on renewable barrier coatings, starch is a promising biopolymer as a raw material.<sup>2</sup> It is abundantly available in high purity and at a low cost and can exhibit thermoplastic behavior. Due to its unique structure starch offers the possibility to numerous chemical modifications to gain desired properties.<sup>3</sup> To overcome

its inherent water sensitivity, several approaches such as chemical substitution, blending with other polymers or cross-linking with poly-functional agents have been studied. Cross-linking is a commonly used technique to increase molecular weight and to enhance e.g. oxygen and water vapor barrier properties in starch-based materials, which is desirable in food packaging applications.<sup>4-6</sup> Citric acid is a food grade tri-carboxylic acid and cross-linking agent and hence used in industrial food applications. Cross-linking reaction with citric acid can occur inter-molecularly between two starch molecules or intra-molecularly within the same starch molecule and even within the same glucose molecule.<sup>7</sup> However, cross-linking between starch and citric acid has been only shown in relation to physical properties like increased gel content and lower water solubility.

During the drying process when producing citric acid containing films or coatings, hydrolysis of starch can occur due to a low pH. The two concurrent reactions, cross-linking and hydrolysis of starch due to citric acid and different techniques to study them are presented in this work. A new approach to detect di-ester of citric acid was developed using complexometric titration with copper(II)-sulfate. Further, analyses of molecular characteristics like molecular weight determinations suggested both cross-linking reaction as well as a simultaneous hydrolysis of starch molecules. We aimed to improve our understanding of the interactions between starch and CA in starch-based films cured at different temperatures, in order to facilitate processing of modified starch for industrial production of accessible films or coatings for food packaging and to better understand barrier properties such as diffusion coefficient and moisture content reduction with CA addition shown in a previous study.<sup>8</sup> This study is part of a larger collaboration where also barrier properties and morphological properties were determined.

## RESULTS

Our study shows suitable methods for detecting cross-linking of starch by citric acid and discusses how citric acid concentration and curing affects the molecular structure of starch. We showed that both cross-linking as well as hydrolysis occurred in citric acid containing solution-cast starch films. We used two methods to detect cross-linking of starch by CA. Weight average molecular weight,  $M_w$ , determinations showed that with increasing citric acid content  $M_w$  decreased only slightly but was enhanced at high temperature drying, 150 °C, resulting in  $M_w$  decreases up to 97%. The determination of the amylose and amylopectin distribution showed that large amylopectin molecules were degraded. The acid hydrolysis of starch evaluated by the determination of amylose and amylopectin distribution were strongly correlated with the measured  $M_w$  ( $r^2=0.923$ ). By comparing the  $M_w$  before and after de-esterification with NaOH, cross-linking of starch by citric acid can be proven. Cross-linking reaction can increase  $M_w$  due to covalent ester-bonds of one citric acid molecule to two starch molecules which, however, can be broken by NaOH resulting in lower  $M_w$ . However, only the water-soluble starch content could be studied by this method. The  $M_w$  of starch films containing citric acid showed a 10-85% decrease after NaOH-treatment. This evidently proves intermolecular cross-linkages in starch by citric acid in all starch films, even in the non-cured films. The decrease in  $M_w$  was largest for the starch films containing 20 and 30 pph citric acid cured at 150 °C. In addition, most films containing citric acid cured at 150 °C had higher  $M_w$  in the water-soluble starch film part than in 0.1 M NaOH which indicates the cross-linking between starch molecules since only cross-linking reaction can increase  $M_w$  of starch.

For the first time, the amount of di-esterified citric acid was measured as the difference in CA titration for hydrolyzed and non-hydrolyzed starch films and was expressed as both a percentage of total citric acid and as degree of di-esterification, DDE, of starch. In all citric acid containing starch films, di-esters were present in concentrations ranging from 2.2 to 21.3% of total added citric acid. The highest concentration of CA, 30pph,

gave the highest DDE, ranging from DDE 0.008 (every 125<sup>th</sup> anhydroglucose monomer unit being di-esterified) up to DDE 0.054 (every 19<sup>th</sup> anhydroglucose monomer unit being di-esterified) for non-cured and 150 °C cured films, respectively. Citric acid concentrations between 5 and 20 pph resulted in lower DDE values of between 0.002 and 0.006. These results do not differentiate between inter- or intramolecular di-esters, as described earlier.<sup>7</sup> However, considering the results from the  $M_W$  measurements in water, with  $M_W$  decreases of 85% after de-esterification, there is clear proof of intermolecular di-esters, i.e. cross-linkages, in the starch films, since intra-molecular di-esters would not cause a reduction in  $M_W$ . We concluded that the 30 pph CA-containing starch films were highly cross-linked, since they exhibited most CA di-ester, highest  $M_W$  decrease after de-esterification and lowest water solubility. The results clearly indicate cross-linkages in starch films without curing at high temperature, which to the very best of our knowledge, have not been achieved or demonstrated by any other study.

## CONCLUSIONS

For the first time, it was shown that no high temperature is needed to cross-link starch with CA. However, the degree of cross-linking increased with higher temperature, at least for films with 30 pph CA. Furthermore, it was seen that starch was degraded during both drying and curing when CA was present and that curing at 150 °C resulted in severe hydrolysis as seen in  $M_W$  decrease up to 97% and the amylose and amylopectin distribution measurements. We believe that this is the first study that detected and quantified CA di-ester using a modification of the complexometric titration with copper (II)-sulfate. The detected degree of di-esterification was between 0.002 and 0.054 for films where CA was added. Furthermore, a method to detect cross-linking with regard to  $M_W$  changes in the starch polymer has been described for the first time showing a reduction of  $M_W$  of 10 to 85% after de-esterification in all CA containing starch films. Moreover, measurements of physical properties such as water solubility, gel content and  $T_g$  confirmed the results of the molecular analyses. The here described CA starch films are an easy way to produce cheap materials for water vapor-barrier coatings for e.g. packaging material without a necessary curing step. The material showed increased water vapor barrier properties and can be used for food contact materials since CA is approved as a food additive.<sup>8</sup>

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### 5.2. Kommer Brunt, Heerenveen (The Netherlands)

Improvement of the AOAC 2009.01 dietary fibre analysis in high starch containing matrices

Applying the AOAC 2009.01 total dietary fibre method, significant amounts of low molar weight dietary fibre (LMWDF) has been established in wheat grain based food products. The dietary fibre content in selected wheat grain based food products without prebiotic fortification have been established with both the classical AOAC 985.29 dietary fibre protocol and with the new AOAC 2009.01 total dietary fibre protocol. There was a good agreement between dietary fibre content measured with the AOAC 985.29 method and the fraction high molar weight dietary fibre (HMWDF) measured with the AOAC 2009.01

method. But with this new AOAC method also a significant amount of low molar weight dietary fibre (LMWDF), ranging from 1 – 3 % w/w, was measured.

In a series of samples the LMWDF fractions were isolated with semi-preparative GPC and subjected to a chemical characterization. Firstly, the isolated LMWDF fractions were checked for the presence of mono, di, and oligosaccharides by high performance anion exchange chromatography with pulsed amperometric detection (HPEAC-PAD). The mono and di-saccharide contents were neglectible (a few samples contain traces ( $\leq 0.1\%$ ) of free glucose). In the HPAEC-PAD complex peak patterns of oligosaccharides were detected. Secondly, the monosaccharide composition of the LMWDF fraction was measured. Therefore the isolated LMWDF fractions were subjected to methanolyses and a TFA hydrolysis, followed by (HPEAC-PAD). In all cases glucose makes up for about 90% of the total monosaccharide content after hydrolyses, except for the samples wheat grain and wheat middlings, which contain lower glucose contents of about 80 and 70 % respectively. But also arabinose, galactose, xylose and mannose were present. And applying a mild acidic hydrolysis with 0.05 M sulphuric acid followed by HPAEC-PAD analyses of the hydrolysates indicated also the presence of significant amounts of fructose containing oligosaccharides. It is known that grains contain small amounts of fructans. The presence of fructooligosaccharides was confirmed with the AOAC 999.03 analysis for fructans. The established fructan contents range from about 0.5 % (bread) up to about 3.5% (wheat middlings).

Additional AMG treatment of the LMWDF fraction proved that most of the glucose which was set free by the methanolysis/TFA treatment originate from residual not fully hydrolysed digestible malto-oligosaccharides which were erroneously measured as LMWDF constituents. This leads to the conclusion that the available starch is not fully converted into glucose and maltose by the pancreatic  $\alpha$ -amylase/AMG enzymatic hydrolysis (first step in the AOAC 2009.01 protocol), but that still some minor amounts of residual malto-oligosaccharides are present which erroneously are quantified as LMWDF. This imperfection in the AOAC 2009.01 method can easily be eliminated by introducing an (second) AMG hydrolysis step in the protocol after evaporation of the ethanol and dissolving the residue in deionized water and before desalting the solution.

### 5.3. **Nikola Sakač**, Osijek (Croatia)

Determination of the botanical origin of starch using direct potentiometry and principal component analysis

Starch is a semicrystalline biopolymer and is stored in various plant locations, such as in cereal grains, roots, tubers, stem piths, leaves, seed, fruit and pollen. Starch, as a low-cost polysaccharide, is the most widely used thickening and gelling agent in the food industry. It imparts texture to a great diversity of foodstuffs, such as soups, potages, sauces, and processed foods. Starch granules in higher plants contain two principal types of polysaccharides: amylose and amylopectin. The starch granules from different botanical sources also vary in size, shape, and content of amylose and amylopectin, which affects their chemical and physical properties.

The typical origin analysis of starch includes indirect techniques that measure the differences in the physical and chemical properties of the starches. The tools often used to identify starch origin include optical and electronic microscopy, enzymology, rheology, chromatography, NMR, X-ray diffraction, viscometer profiles and FTIR spectroscopy.

A new approach for the determination of the botanical origin of starch is presented. The sensory principle was based on the formation of starch–triiodide complexes. The starch samples were extracted from wheat, potato, maize, rye, barley, rice, tapioca and a commercial model starch. The amylose/amylopectin ratios of starches, among various

other properties, differ between starches of different botanical origins. Triiodide ions bind characteristically to the amylose and amylopectin of the starch depending on the starch's origin. The new sensory technique includes direct potentiometric measurements of the response of free triiodide ions in starch–triiodide solutions and principal component analysis (PCA). PCA gave graphical results for statistical differentiation between starches of different botanical origin.

The proposed method is more convenient, simpler, easier, less expensive and more objective for the determination of botanical starch origin than the usual microscopic methods.

#### 5.4. **Shazia Juna, Anton Huber, Graz (Austria)** Fundamental molecular and hydrodynamic characteristics of native starches employing asymmetrical flow-field flow fractionation

Variations in the molar masses and sizes of starches arise due to the differences in botanical source of starch, starch material history (extraction of starch, storage, etc.), dispersion or dissolution method and analytical techniques employed.<sup>1-4</sup> The prerequisites for the accurate determination of the molar masses and sizes for starch are its complete dissolution and the suppression of aggregation of starch macromolecules.<sup>1, 5-8</sup> However, the complete dissolution of starch in aqueous media is not trivial as aggregation of starch macromolecules, arising from both inter- and intramolecular interactions, cannot be completely overcome. Asymmetrical flow field-flow fractionation coupled with multi-angle light scattering and refractive index detector (AF4/MALS/RI) allows the elution and separation of both starch macromolecules and aggregates depending on the flow regimes.<sup>1, 5-11</sup> Separation of molecules and particles in the AF4 channel occurs due to differences in their diffusion coefficients ( $D_T$ ).<sup>10, 12-19</sup> Cross flow rate ( $F_{cr}$ ) is the driving force for inducing separation of molecules/particles (eq. 1).<sup>15</sup>

$$D_T = \frac{(t^0 F_{cr} w^2)}{6 V^0} \frac{1}{t_r} \quad \text{eq. 1}$$

$t^0$  is the void time,  $w$  is the channel thickness,  $V^0$  is the geometric void volume and  $t_r$  is the retention time.

To evaluate the influence of temperature (employed during dissolution process) upon the molar mass and size distributions of waxy corn starch (amylopectin) were evaluated by AF4/MALS/RI.<sup>20</sup> The starch dispersions were heated at various pre-selected temperatures (100°C-200°C) in a dedicated microwave reactor. During the heating process, the temperature, microwave power and pressure were carefully monitored and controlled. The molar masses and sizes of waxy corn starch heated in water did not vary considerably as a function of temperature, indicating that re-aggregation of starch occurs upon cooling to room temperature. However, the corresponding molar masses and sizes of waxy corn starch heated in 1M KSCN were considerably lower than the values obtained in water.

The aggregation of starch macromolecules can be considered as self-assembly of starch moieties and a detailed study on the formation of nano-structures of starch materials was undertaken. Starch is a highly attractive source of nanomaterials as it is highly abundant in nature and biodegradable. Starch nanomaterials were isolated from various botanical sources employing a simple nanoprecipitation technique. A variety of nanomaterials may be designed by considering the mode of treatment (how starch material is treated), botanical source of starch and surface interactions during drying. Temperature employed during the alkaline treatment of waxy corn starch upon the resultant shapes and sizes of waxy corn nanostructures is shown in Fig. 1.

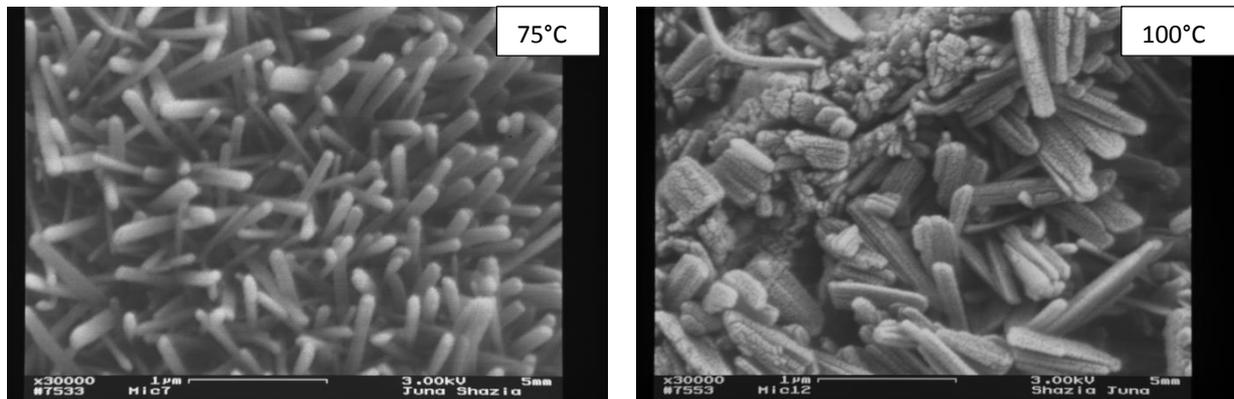


Fig. 1: Influence of temperature upon the shapes and sizes of waxy corn nanostructures.

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## 6. Sweeteners

### 6.1. Tom Kleinhout, Leiden (The Netherlands) New enzymes for the production of specialty syrups

Most common products in the current carbohydrate processing market are glucose (DP1), fructose and maltose (DP2) syrups, and maltodextrins (DE 6-20). Nowadays most of the production of these commodity or semi commodity ingredients relies on enzymatic conversions. Alpha-amylases are used in liquefying the starch and for production of low-conversion products; e.g. maltodextrins. Gluco-amylases and beta-amylases are used in saccharification resulting in glucose (or dextrose) and maltose syrups. And glucose-isomerase can be used to catalyse the isomerisation of glucose into fructose; e.g. to make high fructose corn syrup. In the past decade the availability of market products has not changed dramatically, in fact both products and processes have not seen much change

With the right technology the boundaries of the carbohydrate processing market can be pushed into new territories; a technology which combines processing know-how with the right tools. DuPont Industrial Biosciences is expanding its carbohydrate processing portfolio with two new enzymes for specialty syrups. The first is a maltogenic alpha-amylase expressed in *Bacillus licheniformis*. This amylase adds value to carbohydrate processing; due to its broad pH and temperature range, and temperature dependent specific activity it proves to be an excellent enzyme for a variety of maltose applications.

The second product to be presented is a maltotetraose producing amylase from *Pseudomonas saccharophilia* expressed in *Bacillus licheniformis*. OPTIMALT® 4G is able to convert liquefied starch into more than 45 % of maltotetraose, and even more than 60 % when used in combination with a debranching enzyme. Its optimal performance matches industrial standard conditions for maltose production, typically pH 5.0 – 5.5 at a temperature of 60 °C with 32 % DS substrate and DE 10. Maltotetraose syrup has a wide range of applications, among others within food products it prevents hygroscopicity and colouration, it regulates the freezing point, it increases viscosity and the syrup itself has a clean taste and a mild sweetness. This unique enzyme will bring new opportunities for new products for the carbohydrate processing market.

## 6.2. **Michael vom Dorp**, Krefeld (Germany) Composition and applications of glucose Syrups

In the plant world, Nature passes its energy to the next generation through starch, giving the next generation a ready source to feed from as it grows.

The glucose syrups that we make today spring from an industrial heritage that has improved as technology has made possible products that are more finely tailored, purer, to more demanding specifications and supporting a wider area of applications - but what we do today shares many of the original principles that made these products a sound basis for the traditional applications that we know and with which we are familiar.

Our glucose syrups play an important role in providing products that bring enjoyment and fun to the lives of many. Today that heritage is supported by modern processing techniques that allow us to produce a wider and more sophisticated range of profiles and to produce an higher quality of product that, through modern supply chain techniques, arrives at our customers in the best condition for use in their processes.

And with those modern techniques come additional advantages, allowing our syrups to be used in the widest range of ways in existing and new products - giving us the tools we need in order to work with you to deliver the optimal 'sugars' profile that suits your product, processes and formulations.

So, even though glucose syrups have a long and dependable history, we are still learning how to use them in new or improved ways. These ways are not purely sucrose replacement in its own way but more functionality, viscosity, body, mouthfeel, freezing-point-depression, sweetness adjustment, flavor enhancement and nutritional aspects.

The actual discussion about High-Fructose-Corn syrups used in the USA with mainly 55% fructose content should be reduced to the facts which show that mostly products with 9 – 30% fructose are used in Europe which have still significant amounts of higher saccharides.

C\*Sweet glucose syrups are obtained by hydrolysing starch, a process that cleaves the bonds linking the simple sugar units to varying degrees, yielding a mixture of dextrose,

maltose, maltotriose and higher polysaccharides. This composition - the sugar profile – determines the properties of the final syrup. By controlling the concentrations of the constituents, Cargill produces a range of "fine-tuned "C\*Sweet glucose syrups and C\*TruSweet glucose-fructose syrups.

## 7. Starch Applications

### 7.1. **Sabine Genest**, Dresden (Germany)

The removal of stickies with modified starch and chitosan - highly cationic and hydrophobic types compared with unmodified ones

Biologically degradable polymers based on renewable raw materials represent an alternative of considerable importance to synthetic polymers. For this purpose chitin and starch are the most important raw material sources for cationic polysaccharides.

Polymers bearing charged groups, so-called polyelectrolytes, and their hydrophobically modified derivatives are extensively used in both nature and industry. This large applicability is the result of their unique amphiphilic properties and viscometric behavior in aqueous solution. We are actually working on new application fields of polysaccharide derivatives with high degrees of substitution [1-4]. The study of solution dynamics of polyelectrolytes is necessary to gain insight into many biological and industrial processes.

Through alkaline splitting-off of the N-acetylic group (deacetylation) from chitin, one obtains the biopolymer chitosan. Chitosan is a product whose degree of deacetylation (DA) amounts more to than 50 % and is soluble in organic acids. This acidic environment leads to a protonation of the present amino groups, which results in a natural, cationic polyelectrolyte commercially available. Our cooperative working group in Jena realized the synthesis of hydrophobic modified potato starch and chitosan by introducing benzyl groups with different degrees of substitution ( $DS_{Bn}$ ). For the starch samples a one-pot-reaction could be established which allows a contemporary introduction of positively charged groups (molecular degree of substitution,  $MS_N$ ) through reaction between the hydrophobic starch derivate and 2,3-epoxy-propyltrimethyl ammonium chloride. One could obtain a high degree of substitution for both benzyl with  $DS_{Bn}$  0.27 – 0.74 and cationic groups with  $MS_N$  0.23 – 1.0.

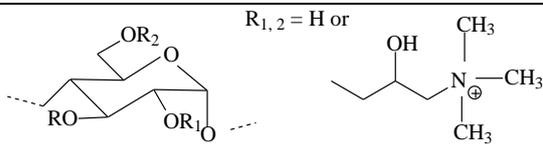
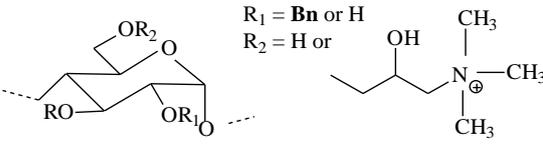
The oral presentation will focus on the one hand on the characterization of such highly substituted polysaccharides concerning their rheological behavior, charge density, dynamic surface tension measurements and particle size distribution to identify correlations to their amphiphilic character. We also compare our results [5] to unmodified chitosan and cationic starch samples to get to know the effect of the introduced hydrophobicity in a systematic way.

On the other hand we present a promising application of these amphiphilic polysaccharides for paper technology, where the effective control of so-called stickies being particles with high adhesive power and causing enormous problems during paper production, is not yet solved. Recently we found that stickies being dissolved and colloidal substances (DCS) in paper cycling water can be removed with tailored natural polymers, wearing cationic as well as hydrophobic groups [3]. For this purpose model suspensions based on dissolved paper pulp of different kind of papers have been prepared and the sticky content has been characterized by turbidity measurements, charge density determination and the total organic carbon content (TOC). The addition of latex particles leads to an increase of sticky amount. For the first time, dynamic surface tension measurements have been established as a useful tool for the characterization of the removal of stickies using tailored cationic, amphiphilic polysaccharides.

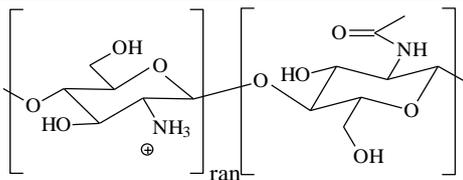
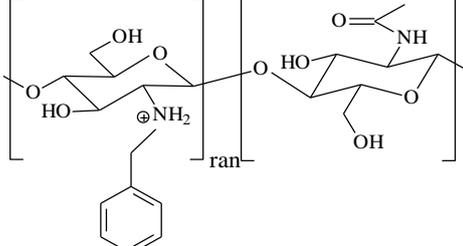
Depending on the properties of the polysaccharide derivatives, two possible mechanisms can be found: “charge dominated removal” or “removal dominated by hydrophobicity”. It seems that turbidity and TOC are lowered especially due to charge interaction whereas the increase of the surface tension is mainly caused by the hydrophobic character of the modified natural polymers.

The following two tables present the polysaccharide derivatives of interest:

(a) starch derivatives

starch sample	structure	MS <sub>N</sub>	DS <sub>Bn</sub>
cationic starch	 <p>R<sub>1,2</sub> = H or</p>	0.28 – 0.67	/
hydrophobically modified, cationic starch	 <p>R<sub>1</sub> = Bn or H R<sub>2</sub> = H or</p>	0.23 – 1.00	0.27 – 0.74

(b) chitosan and its derivatives

chitosan sample	structure	DA	DS <sub>Bn</sub>
commercial chitosan		85 %	/
hydrophobically modified chitosan		85 %	0.50 – 0.67

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Interaction of cationic starch and dissolved colloidal substances from paper recycling, characterized by dynamic surface measurements  
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The removal of stickies with modified starch and chitosan - highly cationic and hydrophobic types compared

7.2. **Petr Duchek, Pilsen (Czech Republic)**  
Starch-Montmorillonit-Composite (Nano-Products)

Starch is a natural polymeric product that is cheap and readily available all over the world. In its thermoplastic form it is one of pillars of biodegradable plastic materials for many years. Principal bottlenecks that hinder this material from its universal use as a biodegradable plastic matrix are low stability in water/hydrophilic environment, and poor mechanical properties.

The first drawback – low mechanical strength – can be improved by addition of different fillers especially from non-metals world. A large variety of both plastic (bentonite and montmorillonite, kaolin) and non-plastic (chalk, silica, diatomite) minerals and rocks has been tested as reinforcement. Especially blending of plastified starch even with low amount of plastic smectic clays (up to 5%wt.) enhances the mechanical strength considerably. Introduction of a small amount of layered silicates into the starch may increase not only the strength of the composite but influence also other properties in a positive way ( e.g. lower flammability, lower gas permeability).

Montmorillonite (MMT) is a three layer aluminosilicate occurring in smectic clays like bentonites. It exhibits a unique structure which is reflected in distinct ion-exchange properties and high surface area (up to 800 m<sup>2</sup>/g). This mineral finds its application in many industrial and agricultural sectors and not by chance is called “a mineral of thousand uses”. A unique mineral microstructure (sheets of about nanometer thickness and hundred – several hundreds nanometers length determines this mineral and clay as one of the most important components used in nanomaterials (Figures 1 – 3):

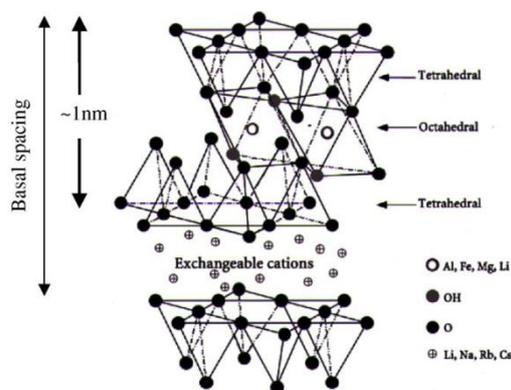


Fig. 1: Montmorillonite mineralogy

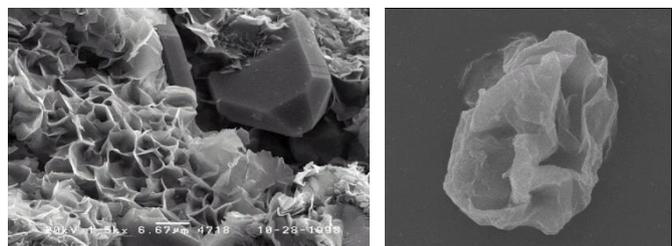


Fig. 2, 3: Montmorillonite microstructure

The interlayer space or the “gallery” accommodates inorganic cations in nature (sodium, calcium, magnesium or mixed structures). These cations are hydrated differently but the overall structure determines the hydrophilic character of this materials and good compatibility with hydrophilic compounds and polymers e.g. plastified starch. Of course, introduction of organic cations (like quaternary ammonium salts) into the gallery by the simple ion-exchange reaction will results in inorganic-organic mixed structures. This organically modified MMT will be then soluble poorly in water and better in polar/non-polar organic solvents.

It was found that different modified starch types (cationized, acetylated, crosslinked) when reinforced by MMT result in composite materials with lower mechanical properties than the pristine potato starch. The results show that MMT (and bentonite, of course) undergoes the disintegration of the mineral microstructure (“house of cards”), and intercalation or even delamination of the mineral. Microscopy (SEM) and X-ray diffractometry prove this structure of nanocomposites.

## 8. Starch Technology

### 8.1. Doris Schieder, Dominik Schwarz, Volker Sieber, Straubing (Germany) Fermentation of potato pulp for production of chemicals

Potato pulp is a by-product of starch production. Today it is mainly used for cattle feed, fertilizer or biogas production. The dry mass of potato pulp contains around 70% (poly)saccharides, which makes it a potential substrate for the production of value

added chemicals by fermentation. However, seasonal production, high water content, and low storage stability impact the utilization of potato pulp requiring adopted strategies for valorization.

We performed polyhydroxyalkanoate (PHA) laboratory fermentation tests with *Curpriavidus necator* spec. as an option for the production of valuable chemicals on potato pulp. Polyhydroxyalkanoates are biodegradable biobased polyesters with physical properties close to polypropylene offering a wide range of potential applications. PHA are synthesized by several bacteria at high carbon substrate levels under limitation of other essential nutrients like nitrogen, phosphor, or oxygen. They are stored within the cytoplasm of the bacterial cells. The storage within the cells offers the possibility to separate the product from the fermentation broth by separating the bacteria cells thus reducing the impact of the broth composition on product separation. Saccharides as well as fatty acids may be used for carbon source matching quite well the chemical composition of potato pulp.

For our experiments we used fresh dewatered potato pulp of 14% dry mass and stored it in evacuated bags at 4-5°C. A mixture of amylases, pectinases, and cellulases, was used to hydrolyze poly- and oligosaccharides and to reduce the swelling capacity of the pulp. Fermentation tests with *Curpriavidus necator* spec. were performed on the hydrolyzed pulp under N-limitation. The selected strain was found to produce around 5 g/l bacterial dry mass in these first tests with PHA (polyhydroxybutyrate) contents of 30% in the cell dry mass consuming glucose as well as acetic acid and lactic acid.

## 8.2. **Johan De Meester**, Vilvoorde (Belgium) The impact of REACH on the starch industry

This presentation will introduce the general scope of the REACH Regulation 1907/2006 as amended when entered into force in June 2007. The most important provisions affecting the starch industry were the effect of the pre-registration and the exemptions on registration of the substance.

Pre-registration for phase-in substances allowed continuing the business in non-food, non-feed and non-pharmaceutical applications until the deadline of registration dictated by the tonnage band.

A number of exemptions to the obligation to registration for substances manufactured by the starch industry will be provided. The interpretation on the difference between substances occurring in nature and intended chemical modification is equally important to provide evidence for exemption on registrations.

If an exemption could not be provided, then registration is needed to continue the business in non-food, non-feed and non-pharmaceutical applications. Products of the starch industry that required registration are wheat glucose syrups, corn steep liquor, non-crystallizing sorbitol and maltitol, ethanol, organic acids as citric acid and gluconic acid. Ethanol and some organic acids are substances requiring classification and labelling under the CLP Regulation 1272/2008 as amended.

Although there are only a limited amount of products that require registration compared to a number of substances produced in other sectors, it seems that the starch industry may have challenges for products that are used to produce chemically modified starches. Manufacturers from these substances could profit from the exemption on chemical safety assessments and chemical safety reports by filing a dossier as transported isolates intermediates under Art.18 of Regulation 1907/2006.

## 8.3. **Götz Kröner**, Ibbenbueren (Germany) Energy supply and energy management in the company KRÖNER STÄRKE

KRÖNER-STÄRKE is an independent and economically sound, family-owned company. Since 1900 the company had made food products in a natural way for the industry and the craft and retail trades. Openly and honestly.

Since the mid 80s KRÖNER-STÄRKE is pre-treating its sewage which derives from the processing of starch in an on-site biogas plant and is using the biogas thereby incurred to produce heat. In 1993 KRÖNER-STÄRKE switched from heavy fuel oil to natural gas as the primary energy carrier for its increased heat demand.

Against the background of an unchanged strong international competition and rising environmental specifications KRÖNER-STÄRKE had and has to face following essential challenges:

- Secure growth in a growing market.
- Reduce production costs: almost 50% of the production costs are energy costs.
- Treat sewage sustainably due to exploding municipal sewage charges.

In the last decades the companies specific consumption and formation of heat, power, fresh water, and sewage could be reduced significantly. Further saving potential is limited.

In 2005 it was decided to build a new bigger energy supply plant and to enlarge the on-site biogas plant in order to ensure the basis of further growth.

The main idea of the project was

- Use of a competitive primary energy carrier which is price stable and reliably projectable.
- The energy carrier should preferably be local and available in the long term.
- The technique used should be of the latest state of the art and highly efficient.
- As far as possible power should be produced and sustainable energy should be used.

Taking into account these criteria brown coal, woodchips, light fuel oil, natural gas and biogas were investigated.

The result was a heat lead combined heat and power (CHP) plant in redundant layout for security reason. Primary energy carriers are brown coal and light fuel oil. The CHP plant can be upgraded to be suitable for the use of natural gas for flexibility reason. The brown coal vessel is combined with a power generation in order to avoid coal tax. The vessel has a combustion capacity of less than 20 MW in order to avoid emission trading.

The biogas deriving from the enlarged biogas plant is used to produce power in order to receive the officially guaranteed feed-in tariff.

The CHP plant built was financed by contracting and is run by the contractor. The contractor acts as a buffer in the view of complex technique and financial risks because of operating trouble.

The biogas plant was equity- and bank-financed and is run by KRÖNER-STÄRKE itself on the basis of many years' experience.







**Thursday, April 25<sup>th</sup> 2013**

**08<sup>30</sup>**

## **Session II**

### **4. Starch Market**

- 4.1. **Harald von Witzke**, Berlin (Germany)  
The End of the Agricultural Treadmill: Prospects for EU Agriculture

### **5. Starch Analysis**

- 5.1. **Carolin Menzel**, Uppsala (Schweden)  
Analysis of citric acid cross-linked starch films
- 5.2. **Kommer Brunt**, Heerenveen (The Netherlands)  
Improvement of the AOAC 2009.01 dietary fibre analysis in high starch containing matrices
- 5.3. **Nikola Sakač**, Osijek (Croatia)  
Determination of the botanical origin of starch using direct potentiometry and principal component analysis

### **10<sup>30</sup> Coffee Break**

- 5.4. **Shazia Juna, Anton Huber**, Graz (Austria)  
Fundamental molecular and hydrodynamic characteristics of native starches employing asymmetrical flow-field flow fractionation

### **6. Sweeteners**

- 6.1. **Tom Kleinhout**, Leiden (The Netherlands)  
New enzymes for the production of specialty syrups

### **13<sup>00</sup> - 14<sup>00</sup> Lunch Break**

- 6.2. **Michael vom Dorp**, Krefeld (Germany)  
Composition and applications of glucose Syrups

### **7. Starch Applications**

- 7.1. **Sabine Genest**, Dresden (Germany)  
The removal of stickies with modified starch and chitosan - highly cationic and hydrophobic types compared with unmodified ones

### **15<sup>00</sup> Coffee Break**

- 7.2. **Petr Duchek**, Pilsen (Czech Republic)  
Starch-Montmorillonit-Composite (Nano-Products)

### **8. Starch Technology**

- 8.1. **Doris Schieder, Dominik Schwarz, Volker Sieber**, Straubing (Germany)  
Fermentation of potato pulp for production of chemicals
- 8.2. **Johan De Meester**, Vilvoorde (Belgium)  
The impact of REACH on the starch industry
- 8.3. **Götz Kröner**, Ibbenbueren (Germany)  
Energy supply and energy management in the company KRÖNER STÄRKE

- 17<sup>00</sup>** **Closing remarks** by the Chairman of the Starch Experts Group, **Willi Witt**, Oelde (Germany)

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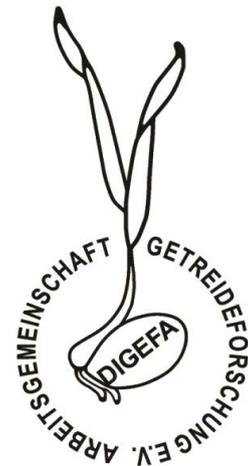


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