



in cooperation with

Max Rubner-Institut
Institute of Safety and Quality of Cereal

and the

University of Hohenheim
Institute of Food Science and Biotechnology

69th Starch Convention
&
14th European
Bioethanol and Bioconversion
Technology Meeting

April 10th – 11th 2018
in Detmold, Germany

Program

Evening Program

Exhibition

Participants

Summaries

Tuesday, April 10th 2018

08⁰⁰ – 08³⁰ Registration

69th Starch Convention

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

1. Raw material

08⁴⁵ 1.1. **Thomas Pruter**, Emlichheim (Germany)
Alternative crops for a traditional potato starch producer

2. Starch

09¹⁵ 2.1. **Qioaquan Liu**, Jiangsu Province (China)
Underlying Reasons for Natural Variation of Starch Biosynthesis in Rice to Improve
Cooking and Eating Quality

09⁴⁵ **Communication Break**

10¹⁵ 2.2. **Sabina Jakobi, Mario Jekle** and **Thomas Becker**, Freising (Germany)
Physical modification of flour - distinction between mechanically and thermally induced
alterations

3. Modification

10⁴⁵ 3.1. **Jung Sun Hong**, Gyeonggi-do (Korea)
Derivatization patterns among chain populations in granular starch by ion-exchange c
chromatography using a fluorescent substituent

11¹⁵ 3.2. **Ali Marefati**, Lund (Sweden)
Starch Pickering emulsions for encapsulation of hydrophobic and hydrophilic bioactive
compounds

11⁴⁵ 3.3. **Fresia Alvarado Chacon**, Wageningen (The Netherlands)
Structure and rheological properties of starch-PE compounds in film blown
applications

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13³⁰ 3.4. **D.C. Saxena**, Punjab (India)
Novel Starches: Comparative studies for physico-chemical, pasting, thermal,
morphological, rheological and structural properties and the development of various
food products thereof

14⁰⁰ 3.5. **Jens Buller, Christina Gabriel, Jana Kolbe** and
Waltraud Vorwerg, Potsdam (Germany)
Starch-based wood adhesives

4. Application

- 14³⁰ 4.1. **Stijn Reyniers**, Heverlee (Belgium)
Impact of multivalent ions on the gel forming capacity of potato (*Solanum tuberosum*) flakes and on texture and fat content of snacks made thereof

15⁰⁰ Communication Break

- 15³⁰ 4.2. **Ruud van der Sman**, Wageningen (The Netherlands)
Swelling of starch microgels in sugar solutions
- 16⁰⁰ 4.3. **Koen Venema**, Maastricht (The Netherlands)
The effects of starch and starch derivatives on the gut microbiota in health and disease”

5. Enzymes

- 16³⁰ 5.1. **Bart Koops**, Leiden (The Netherlands)
A novel enzyme to improve wheat starch and gluten separation.
- 17⁰⁰ 5.2. **Nina Schrögel-Truxius**, Darmstadt (Germany)
Application of arabinoxylan degrading enzymes and their effects in wheat flour process”
- 17³⁰ **Exhibitor’s Forum** – short term presentations
1. Dr. Hongben Zhou, AVA GmbH & Co. KG
Reacting and processing of starch
 2. Jochen Sprung, Andreas Weissenberger, Coperion GmbH.
Innovative solutions for extrusion and bulk solid handling in the starch industry

Wednesday, April 11th 2018

6. Technology

- 08³⁰ 6.1. **Benjamin Metz**, Vienna (Austria)
Starch sweetener plant design – practical experience
- 09⁰⁰ 6.2. **Laurent David**, Nancy (France)
Using modeling to compare and optimize continuous chromatographic processes
- 09³⁰ 6.3. **Dietmar Schulze**, Wolfsburg (Germany)
Silo design for flow – Application on starch silos
- 10⁰⁰ 6.4. **Dominik Wüst**, Stuttgart (Germany)
Hydrothermal conversion of starchy residues – Platform chemicals and char from old bakery goods

10³⁰ Communication Break

Evening Program

Monday, April 09th 2018

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

Buffet

Ceasars salad with dressing

Chicken-sliced fresh mushrooms

Pork-neck-roastmeat with pepper sauce

Potato gratin with bacon and cheese

Brussels sprouts with sauce Béarnaise sauce

Emperor vegetables, rice

Dessert: Vanilla mousse + strawberry sauce

Tuesday, April 10th 2018

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

Wine

Franconia

2017er „Bacchus fünf Freunde“ Weißwein
Gutsabfüllung Zur Schwane, Volkach
Qualitätswein

2017er „Pinkfein“ Rosé (Rotling)
Gutsabfüllung Zur Schwane, Volkach
quality wine

Moselle

2016er „Urgestein“ Erdener Treppchen Riesling Kabinett
Weingut Andreas Schmitges, Erden
Prädikatswein , dry

Palatinate

2015er „Wintersemester Master“ Rotwein
Gutsabfüllung Reinhard Studier, Ellerstadt
quality wine, dry

Rhinehessen

2013er Bechtheimer Spätburgunder
Weingut Spiess, Bechtheim
quality wine, dry

Wuerttemberg

2015er Lemberger Rotwein
Weingut Rolf Heinrich GbR, Heilbronn
quality wine



20⁰⁰ **Social gathering** at the restaurant “Strates Brauhaus”
in 32756 Detmold, Lange Straße 35

Lunch

Lunch will be served in the exhibition hall:

The menu:

Tuesday, April 10th 2018

Lentil stew with smoked little things

Onion soup with croutons

Canapes with herb cream cheese

Canapes with salami

Canapes with salmon

Canapes with camembert

Dessert: Mousse au Chocolat

Wednesday, April 11th 2018

Asian vegetable soup

Party soup with a lot of content

Teriyaki chicken skewers

Tomato and mozzarella skewers

Chicken - plate with mini meatballs

mini baguette rolls

Dessert: Panna cotta with a lot of strawberry sauce

Beverages:

Mineral water

Coca-Cola

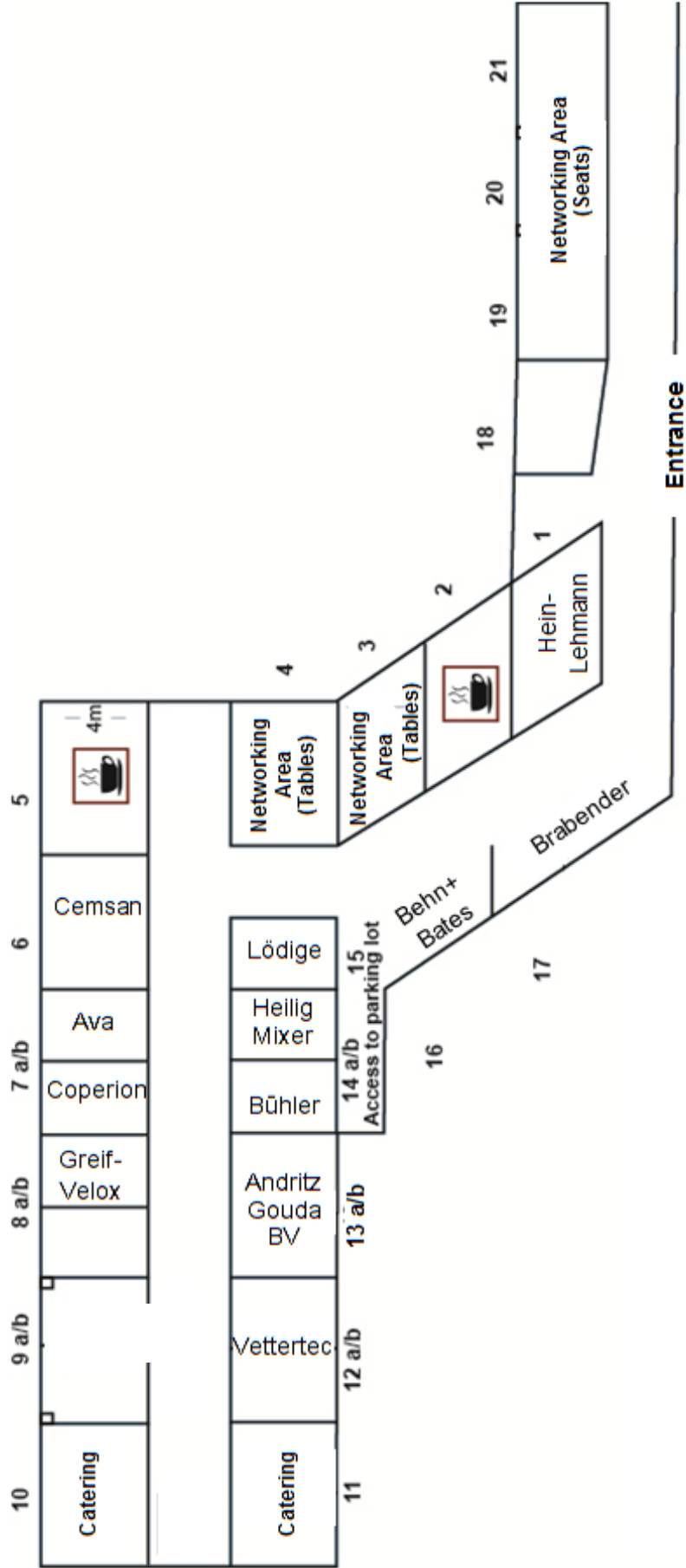
Orange juice

Apple Spritzer

**Bon appétit
and interesting conversations!**

Exhibition Hall Association of Cereal Research Stand allocation

69th Starch Convention and 14th Bioethanol and Bioconversion Technology Meeting from
April 10th – 11th 2018



Exhibition

Andritz Gouda B.V., PD Waddinxveen Netherland

AVA GmbH & Co. KG, Herrsching

Behn + Bates Maschinenfabrik GmbH & Co. KG, Münster

Brabender GmbH & Co. KG, Duisburg

Bühler GmbH, Braunschweig

Cemsan DIS TIC. A.S., Arifiye, Sakarya, Turkey

Coperion GmbH, Stuttgart

Greif-Velox Maschinenfabrik GmbH, Lübeck

HEIN, LEHMANN GmbH, Krefeld

Gebr. Lödige Maschinenbau GmbH, Paderborn

N.M. HEILIG B.V., Heerhugowaard, Netherland

VetterTec GmbH, Kassel

Participants

Effective April 6th, 2018

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Summaries

1. Raw material

11. **Thomas Pruter**, Emlichheim (Germany)
Alternative crops for a traditional potato starch producer

Detailed information will be presented at the convention.

2. Starch

- 2.1. **Qioaquan Liu**, Jiangsu Province (China)
Underlying Reasons for Natural Variation of Starch Biosynthesis in Rice to Improve Cooking and Eating Quality

Detailed information will be presented at the convention.
- 2.2. **Sabina Jakobi, Mario Jekle and Thomas Becker**, Freising (Germany)
Physical modification of flour - distinction between mechanically and thermally induced alterations

Starch is one of the most important biopolymers worldwide determining the functional characteristic of fluid and solid products. During the production of baking products, water from the surrounding dough matrix is bound by starch polymers enabling the formation of a viscoelastic crumb structure. Thereby, the amount of bound water in occurring isothermal and heating processes affect the resulting product quality. In general, those properties are referred to the origin of starch and the environmental conditions during growth. However, the type and extent of grinding significantly alters the hydration of starch and starch-protein matrices, as flours. Depending on the grinding process, equal cereals can show differences in the water retention capacity and thus influence the quality of food products.

The knowledge of starch modification could enable a targeted application of the grinding process in order to achieve a desired functionalization of the flour. However, since mechanistic relations of hydration properties to structural alterations of starch are still not understood in detail, a targeted modification of starchy matrices failed. Therefore, the aim of this study was to identify starch structures altering the hydration behavior of wheat flour (SRC, AACC 76-11) and thus matrix functionality. Furthermore, effects of applied forces during the grinding process – mechanical, thermo-mechanical and thermal – were investigated to clarify the role of thermal forces during grinding on flour structures.

To evidence clear allocation of rise in hydration properties to specific changes in starch structures, specific grinders were used to cause a variety of variations on different structural levels of starch. The impact grinder (IG) is known to have a major effect on particle size distribution caused by impact and shear forces. Beside the mechanically induced flour alterations, modifications could occur due to heat development during grinding. To exclude heat induced alteration, a cryogenic grinder (CG) was used. To prove a (causal) relation, an existing high correlation of structural level and rise in WRC for both grinding methods was required. For IG and CG modified flours, no linear correlation for maltose content (HPAEC-ED, Dionex, Germany) or relative amylose content (assay kit, Megazyme International, Ireland) and WRC was detected. Although for cryogenic grinding a linear correlation of WRC and decrease in gelatinization enthalpy (DSC, Perkin Elmer, USA) was noticed ($R^2 = 0.84$), this correlation could not be proven for UCG modified flours ($R^2 = 0.03$), respectively. Since one type of grinder doesn't confirm a dependency of WRC and reduction gelatinization enthalpy, changes in starch crystallinity did not cause rise in WRC. Therefore, such correlations are misleading when used for interpretation of rise in hydration properties.

The comparison of both grinders confirms variations in starch structures by different physical forces. For a detailed investigation, specific physical stresses (mechanical, thermal, mechanical-

thermal) were applied on pure wheat starch, wheat flour and wheat protein (gluten). High starch-protein interactions causing changes in the gelatinization onset of the wheat flour by thermal stress, which were not detectable for pure starch, were disclosed. This investigation shows that starch and protein structure and consequently functionality depends - beside mechanical forces - to a high extent on thermally induced modifications during the grinding process. Thereby, protein functionality is mainly altered by heat exposure. Gained knowledge promotes the unraveling of structure-function relations of modified flours relating to hydration properties and establishes the basis for a targeted functionalization of flours during grinding.



Sabina Jakobi studied at the University of Hohenheim and holds a M.Sc. degree in Food Science and Engineering. She currently aims for a doctoral degree at the Institute of Brewing and Beverage Technology at the Technical University of Munich, where she works as a Research Associate. She focusses on the impact of mechanical forces on starch structures and resulting changes of flour functionality.

3. Modification

3.1. **Jung Sun Hong**, Gyeonggi-do (Korea)

Derivatization patterns among chain populations in granular starch by ion-exchange chromatography using a fluorescent substituent

Detailed information will be presented at the convention.

3.2. **Ali Marefati**, Lund (Sweden)

Starch Pickering emulsions for encapsulation of hydrophobic and hydrophilic bioactive compounds

Ali Marefati^{1, *}, Maria Matos², Marilyn Rayner¹

1- Department of Food Technology, Engineering, and Nutrition, Lund University, Lund, Sweden

2- Department of Chemical and Environmental Engineering, University of Oviedo, Oviedo, Spain

There is a growing interest in controlled and targeted delivery through structural approaches of emulsions based systems in food and pharmaceutical industries. Starch granules have recently been used to create particle stabilized, i.e. Pickering, emulsions. Higher stability of Pickering emulsions, compared to surfactant and protein stabilized systems, makes them more suitable for encapsulation and delivery systems. This can be attributed to irreversible adsorption of solid particles due to their large sizes resulting in higher energy barrier compared to other stabilizing agents. The objective of the present study has been to investigate the possibility of encapsulation, protection and controlled release of hydrophobic and even hydrophilic substances (in context of multiple emulsions) through starch Pickering emulsions. Moreover, upon exposure to heat, starch granules partially gelatinize and form a cohesive barrier at the oil-water interface.

Curcumin and carmine have been proposed as encapsulated substances. Octenyl succinic anhydride modified quinoa starch was added as the solid particle stabilizing agent. Phosphate buffer and Miglyol 812 were used as water phase and oil phase. Emulsions were created using a high-shear homogenizer. The size and shape of the emulsions were characterized using a Particle size analyser and bright field microscopy respectively and the encapsulation properties were characterized using spectrophotometer. Storage stability of samples was evaluated over 24 h in excess of water for curcumin and 4 weeks for carmine as stored in room temperature. To see the effect of starch gelatinization, some samples were then heated. The encapsulation stability was also evaluated using an in vitro model. Starch stabilized emulsions showed high physical and encapsulation stability both in simple and double emulsions. High stability was obtained also in heat treated samples as a result of the highly stable layer formed by partially

gelatinized starch. Overall, this study demonstrates that Pickering emulsions stabilized with quinoa starch granules have a potential for effective delivery of bioactive compounds.



Ali Marefati

3.3. **Fresia Alvarado Chacon**, Wageningen (The Netherlands)
Structure and rheological properties of starch-PE compounds in film blown applications

Polyethylene (PE) is known for its excellent waterproof properties, but it is permeable to oxygen and other gases. Starch is a good oxygen barrier, but is not water resistant. A good mixture of both materials should result in products with high water and oxygen barrier. The combination of PE and starch in a blend is currently not used for the production of films, but is potentially very interesting. An important advantage is that starch is produced from renewable raw materials such as potatoes, making the film partially bio-based. Gradually bio-based films can replace the existing petroleum-made films.

The present research focus in two main questions:

- How the rheology of a system containing polyethylene and thermoplastic starch can help us understanding the blend structure?
- How to measure the rheological properties of the blend components such that they are representative for the blending process and film structure?

Different methods have been applied in the literature to correlate the film blowing properties and morphology of thermoplastic starch based blends to basic rheological properties like shear viscosity and elongational properties [1]. However, the application of these methods to samples where the amount of water varies was not yet studied. Also, it has been shown in recent publications that elongational viscosity can give an indication of the co-continuity window of immiscible polymer blends and that strain hardening (in one or both components of the blend) expands these window of co-continuity [2]. In a blend where starch is present more parameters such as water content, presence of other plasticizers and plasticizer content play a crucial role. In practice, the main challenge is preventing that water evaporates during analysis and that analysis conditions are similar to the processing conditions.

There are different methods to measure the rheology of starch. In this study, three methods were used to characterize the rheology of starch: A rapid visco analyzer, a capillary and a dynamic rheometer. Each method provides different information that could eventually be correlated to the blending process or to the blend structure.

By using these methods the effect of glycerol as plasticizer in the rheology of the system was studied, results could be related to the extrusion process. Also, the effect of starch types with different rheology was studied and related to the morphological structure of the blend.

[1] Zullo, R and Iannace, S. The effects of different starch sources and plasticizers on film blowing of thermoplastic starch: Correlation among process, elongational properties and macromolecular structure. *Carbohydrate Polymers* 77 (2009) 376–383

[2] Hedegaard et al. Effect of extensional viscosity on cocontinuity of immiscible polymer blends. *Journal of Rheology* 59, 1397 (2015) 1397-1417.



Fresia Alvarado Chacón, *phD*, after finishing my *phD* in physics in 2007 I started my career in research and development of plastic products. I developed aramid non-wovens for a Dutch company and then shifted to bio-based products. In 2015 I completed an education on polymer/plastics engineering and since then I work for Wageningen Food and Biobased Research doing research and project leading of sustainable plastics.

3.4. **D.C. Saxena**, Punjab (India)

Novel Starches: Comparative studies for physico-chemical, pasting, thermal, morphological, rheological and structural properties and the development of various food products thereof

Most of the world population receives their nutrition from three cereal species like wheat, rice, and maize. To extend the food base, we should not neglect lesser-known indigenous crops, such as Amaranth spp., Chenopodium spp., Horse Chestnut, Waterchestnut, Sweetpotato, Dolichos uniflorus, Moringa oleifera etc. In the present study, the starches from various non-conventional sources were isolated and characterized for their physico-chemical, pasting, thermal, morphological, rheological and structural properties. Also, different food products were developed from these starch sources and their specific qualities were compared which could be useful for industrial purposes.

Among them amaranth (*Amaranthus* spp.) is a highly nutritious crop and could rise to universal prominence as a food crop. Smaller granules of amaranth have a greater water-binding capacity, higher swelling power, lower gelatinization temperature and high resistivity to amylases. Amaranth starch shows good gelatinization properties and freeze/thaw stability appreciated in the food industry. The SEM micrograph of raw amaranth flour exhibited intact starch granules embedded in a very dense protein matrix. Conversely, starch granules had been degraded in the germinated amaranth flour. The results of pasting profile revealed that germination significantly affects pasting properties of amaranth flour as it decreased the values of peak viscosity, breakdown, setback, final viscosity and pasting temperature. Germinated amaranth flour showed substantially lower peak (93.32 RVU), trough (62.58 RVU), breakdown (10.66), final viscosity, (82.33 RVU), setback (12.16 RVU) than that of raw amaranth flour (124.26, 98.0, 26.0, 115, 17.6 RVU), respectively.

The *Chenopodium album* plant is widely distributed in Western Rajasthan, Kulu valley, Shimla, Mumbai, Kashmir, and Sikkim acting as the prominent cultivators of the crop within India. *C. album* is considered a functional food grain as it exhibits antioxidants, polyphenols, flavonoid as phenolic amide & glycosides (quercetin, rutin, kaempferol), saponins, cinnamic acid amide, alkaloid chinoalbicin, apocortinoid, xyloside, lignans and carotenoids as active phytoconstituents and hence possesses the enormous potential of lowering the risk of various diseases. *C. album* is known to produce cereal like starch-rich seeds and has gained popularity nowadays due to its adaptability and ability to grow under conditions normally inhospitable to other grains. The main constituent of *Chenopodium* grain is the small sized (<1 μm) starch granule with an amylose content of about 11 % and being a major constituent of grains, affects the functional and physico-chemical properties of starch, including its pasting, gelatinization, retrogradation and swelling characteristics. The starch showed lower swelling power, pasting temperature and gelatinization temperature while higher water and oil binding capacity, pasting viscosities and solubility than commercially available starches. Starch showed polygonal shaped granules, dominant elastic behavior and formation of firmer gels with higher hardness, adhesiveness and cohesiveness values for *C. album* V2 than *C. album* V1 starch. Morphological examination of the starches showed the varying size of starch granules with average granule size of 1.021 μm and 1.033 μm found in *C. album* V2 and *C. album* V1, respectively. Microscopic observations of the *Chenopodium* starch reveal the established organization of starch granules in form of clusters. The X-ray diffraction pattern of *Chenopodium* starch displayed "A" type diffraction pattern with peak intensities observed at 15.23, 17.13, 18.19 and 23.32° that are comparable with cereal starches.

Indian Horse Chestnut or Himalayan chestnut (*Aesculus indica*), locally known as Han dun belongs to shady ravines of Jammu and Kashmir, Himachal Pradesh and Uttar Pradesh. The seeds are about 3.5 cm in diameter with a hard shining black rind from outside and lime white cotyledons inside that are mainly consumed by wild animals in hilly areas while in plains the seeds have no use as such and mostly go waste. Moreover, seeds also have edible uses and have been consumed as food during famine times by various tribes of North and North Eastern India. The seeds also possess medicinal properties, used to cure fevers, piles, wound healing, viral infections, rheumatism, skin diseases and cardiovascular diseases. The seeds constitute about 50.5% moisture, 5.85% sugars, 0.39% protein, 1.93% ash and about 38.3% starch on dry weight basis.

Thus, Horse Chestnut starch can be a better option for starch substitution in products and to meet the industrial demand for starch as starch is widely used in food and non-food industry as encapsulating agent, stabilizer, binder, thickener and gelling agent. However, the native starch has limited application and in order to extend its application, the starch has to be modified. Starch modification involves the alteration of physical and chemical characteristics of native starch to improve its functional characteristics that can be used to tailor starch to specific food applications. Modification of starch is carried out to generate novel starches which have new functional and value added properties as demanded by the industry. Various approaches are now available to modify the starch including physical, chemical and enzymatic methods.

The particle size distribution of Horse Chestnut flour has revealed larger particle size of HCN flour with average particle size reported as 0.459 mm. Horse Chestnut seed flour was analysed for physicochemical, functional, pasting and thermal properties. Horse Chestnut seed flour showed 93.28 L* value, 3.05 a* value and 12.97 b* value. Horse Chestnut seed flour was having moisture content of about 10.71%. Crude fat, crude fibre, protein, ash and starch content of Horse Chestnut seed flour were found to be 3.27, 6.34, 5.78, 4.16, and 42.19%, respectively. Carbohydrate content of Horse chestnut seed flour calculated from difference method was 69.74. Calculated energy value for Horse Chestnut seed flour was 331.51 Bulk density of Horse chestnut flour was 0.64 g/ml. True density and porosity of Horse chestnut seed flour was 1.25 g/ml and 48.80%.

The water binding and oil binding capacity of Horse Chestnut flour was 2.21 g/g. and 3.15 g/g, respectively. The foaming capacity and stability of Horse Chestnut flour was 20.20% and 22.32%, respectively. Horse Chestnut flour showed emulsion activity of 58.69% and emulsion stability of 62%. The initial gel formation for Horse Chestnut seed flour occurred at concentration level of 4 g/100 ml while sedimentation volume was observed to be 25ml. Pasting temperature (PT) for Horse Chestnut seed flour was 66.05 °C, which depends on resistance of starch to swelling. Peak, trough, breakdown, setback and final viscosity of Horse Chestnut seed flour was 505 cP, 354 cP, 151 cp, 118 cP and 472 cP, respectively. The gelatinization temperatures including onset, (To), peak, (Tp) and conclusion (Tc), for Horse Chestnut flour were 60.12, 69.90 and 81.53 °C, respectively.

Isolation of starch from Horse Chestnut seed flour was carried out using alkaline steeping process, where alkali (NaOH) concentration was varied from 0.1-0.3% w/v while steep-time was varied from 3-12 h. Starch yield and purity (in terms of protein fraction) were two important factors taken into consideration for process standardization for isolation of Horse Chestnut seed starch. 0.25% alkali concentration and steeping time of 9 h were observed as optimum conditions for extraction of starch from Horse Chestnut seed flour.

Native Horse Chestnut seed starch isolated under optimum conditions was analyzed for physico-chemical, pasting, thermal, morphological and structural properties. The starch showed high degree of whiteness with L* value of 96.2. Moisture, protein, and ash content of native starch were 9.17%, 0.31% and 0.29%, respectively. Native starch showed 26.10% amylose content, 0.85 g/g water binding and 0.65 g/g oil binding capacity. Maximum values of swelling power and solubility of the starch was obtained at higher temperature. Light transmittance of the starch paste showed a gradual decrease with increase in storage period. Pasting characteristics including peak, trough, breakdown, setback and final viscosity were 4110 cP, 2458 cP, 1652 cP, 1369 cP and 3827 cP, respectively. Hardness, adhesiveness, cohesiveness and gumminess of

native starch gel were 0.85N, 0.877Ns, 0.53, and 0.45N, respectively. The native starch granules were round, irregular, and elliptical in shape with smooth surface. Gelatinization temperatures including onset, peak, and conclusion for native Horse Chestnut starch were 53.35 oC, 58.81 oC and 63.57 oC. The native HCN starch showed typical C-type X-Ray diffraction pattern with characteristics peaks at 5.7, 15, 17.30, 18.87, 23.3 and 26.39°.

Water chestnut (*Trapa natans*) belongs to the family Trapaceae, one of the free floating plants, grown in shallow water fields, ponds or swampy lands in tropical and sub-tropical countries. The interesting feature of water chestnut is the color and shape of its outer cover in which the kernel is encased. The water chestnut meat is covered with a thick jet-black outer pericarp shaped like a horn protruding from the head of a buffalo. The outer pericarp is hard, making it quite difficult to peel off to obtain the internal white fruit. Due to the sweet, tender and delicious taste, cooked water chestnut is one of the popular starchy desserts in Asian countries. The nuts are also known for their medicinal importance. Water chestnut is grown throughout India and very popular commodity especially among rural regions. The red variety (lal) widely grown in regions around Punjab, Haryana etc. is popular among masses.

Water chestnut starch contained 0.18% of protein, 0.22% ash and 0.11% lipids, thus giving starch of 99.49% purity. Water chestnut starch exhibited lower water binding capacity (88.5%). The swelling power in water chestnut was lowest at 9.72 g/g. Water chestnut starch granules were observed to be smooth, oval to irregular or cuboidal. The size of granules present in water chestnut was found out to be ranging from small to medium (5- 30 µm). Most of water chestnut starch granules were found out to be smooth but some had slight wrinkles.

The comparison of different properties of different novel starches clearly exhibited the specificity of the particular source and provides a potential to utilize these starch sources on commercial scale to boost economy of the particular region where these are grown.



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1. Former Head of Department of Food Engineering & Technology
2. Former Dean (Planning & Development)
3. A visiting Faculty of Asian Institute of Technology, Bangkok (Thailand).
4. Chairman of the Mentor Council for Food Processing & Preservation Sector under Ministry of Labour and Employment, Govt. of India.
5. Member of Editorial Board of Journal of Food Science and Technology for many years.
6. Member of various Selection Committee

3.5. **Jens Buller, Christina Gabriel, Jana Kolbe and Waltraud Vorweg**, Potsdam (Germany) Starch-based wood adhesives

Starch based adhesives are industrially used such as in the production of corrugated board or paper bags, as well as in purposes like bottle labelling, wallpaper decoration or glue sticks. A lot of optimized starch and dextrin products are available on the market for the adhesive application in the packaging industry on paper basis.

Another situation can be obtained in the field of wood bonding. Predominately synthetic adhesives are applied in wood materials like furniture, floors or in other indoor building products. Technical solutions are still needed to increase the bonding strength of starch-based adhesives. The Fraunhofer institutes IAP and IFAM are following the aim to explore the potential of starch derivatives as wood adhesive with industry partners in a common project. Dependencies of wood bonding strength on chemical structure and physical properties of starch propionates have been investigated in the first step taking into account the following application criteria:

- water dispersibility
- concentration > 30%

- technical processability regarding viscosity and cohesion
- sufficient bonding on selected kind of wood

The synthesis of pea starch propionate samples was combined with different starch degradation methods. High molecular starch propionate was degraded by α -amylase in the first route; oxidized starch was esterified in the second route. Molar mass was adjusted in the range of 105 - 106 g mol⁻¹. Water solubility of more than 50 % was achieved, partly by thermal treatment at 90°C. Aqueous dispersions were mostly prepared with the concentration in the range of 30-45%. The adhesive properties were characterized by measurement of the tensile shear strength with beech wood according to the standardized test DIN EN 204.

Samples of both synthesis routes passed D1 criteria with values of tensile shear strength closed to 10 MPa. The best results with highly concentrated aqueous dispersions were obtained by samples with d.s. of 0.2 – 0.5 in the range of Mw between 4 - 8·10⁶ g mol⁻¹ under the precondition of high water solubility. The viscosity amounted to 103 – 105 mPa·s⁻¹.

Within the investigations also industrial test products were identified which were used in an industrial application test for veneer bonding. Tensile tests of the veneers showed good bond strength.

The higher criteria D2 could be only passed by suitable additives, which strongly influence the hydrophilic properties of the formulation of starch propionate.



***Dr. Jens Buller**, born 1982, is a chemist and received his PhD in polymer chemistry at university of Potsdam. Since 2013 he is working as a scientist in starch modification at the Fraunhofer Institute for Applied Polymer Research. Since 2017 he is head of department Starch Modification / Molecular Properties. His main interests in research lie in modification of starch particularly for technical applications like paper, adhesives and plastics.*

4. Application

4.1. **Stijn Reyniers**, Heverlee (Belgium)

Impact of multivalent ions on the gel forming capacity of potato (*Solanum tuberosum*) flakes and on texture and fat content of snacks made thereof

Dehydrated potato derivatives are used for producing potato-based convenience foods such as instant mashed potatoes and deep-fried potato crisps. These derivatives are made by boiling potatoes after steam peeling, mashing and subsequent drying. The resultant products are then sold as drum-dried potato flakes (PFs) or as air-dried potato granules. They contain cold water swelling starch which readily develops viscosity upon hydration. PFs have a substantial portion of broken cells (40-60%) and extracellular starch (E-S). These characteristics are believed to determine the functionality of PFs during processing. An important feature is that, in contrast to what is the case for cereal starches, potato starch contains phosphate groups on its amylopectin. This leads to rapid hydration, swelling, and high viscosity development during heating of PF suspensions. We here evaluated the impact of (multivalent) ions on the physicochemical properties of commercial PFs [80% starch, 8.0% protein, 3.0% ash and 7.0% dietary fiber; all on dry matter (dm) basis] and their role during the production of restructured potato-based crisps.

Adding NaCl, CaCl₂ and AlCl₃ (125 μmol cation/g PF dm) to suspensions containing 0.25% (w/v) PF reduced their swelling power by 10% (NaCl) and 30% (CaCl₂ and AlCl₃). The same dosages used when studying instant viscosity development in a Rapid Visco Analyzer (RVA) model system (8.0% dm PF, total sample size 25.0 g), reduced the peak viscosities by 5% (NaCl) and 20% (CaCl₂ and AlCl₃). Di and trivalent cations do not only shield negative charges of the phosphate monoesters on the starch chains, they can also form bridges between two phosphate groups of adjacent amylopectin molecules and thereby reduce hydration and swelling even further. Di and trivalent ions had a similar impact on PF functional properties.

Shielding (by Na⁺ ions) or bridging (by Ca²⁺ and Al³⁺ ions) of phosphate monoesters on neighbouring amylopectin chains was also accompanied by a significant decrease (5 and 25%) in E-S content, respectively. Separation of the extracellular amylose and amylopectin fractions by High Performance Size Exclusion Chromatography (HPSEC) showed that adding multivalent ions to PF suspensions mainly decreased the extractability of amylopectin. Although the E-S content decreased by about 25% upon addition of Ca²⁺ ions to the PF suspensions, cold paste viscosity values in the RVA model system significantly increased from 744 to 1,029 mPa.s. Multivalent ions can thus improve the gelation properties of PF suspensions by impacting on amylopectin cross-linking. In a next step, PFs (400 g) were mixed with deionized water (300 g) and (mono and diacylglycerol based) emulsifier (4.0 g) into a crumbly dough that was subsequently laminated into a continuous dough sheet. The latter was then cut into oval shaped pieces which were deep-fried in sunflower oil at 180 °C for 12 sec. Including Ca²⁺ ions (125 µmol/gdm) in the dough recipe significantly increased the specific strength of the dough sheet (from 1.47 to 1.62 N.cm³.g⁻¹). Low-field Proton Nuclear Magnetic Resonance analysis of dough sheets showed that amylopectin cross-linking significantly lowered the mobility of protons in the starchy gel network (relaxation time T_{2E} decreased from 6.15 ms to 5.42 ms) without affecting amylose crystallization providing direct evidence that the Ca²⁺ ions strengthen the gel network of the sheeted dough. The specific hardness of the final crisp increased by about 10% upon Ca²⁺ addition while the crispness remained unaffected. At the same time, the fat content of the deep-fried crisps was lowered with about 5% of its initial content. We here conclude that multivalent ions can be used to improve the pasting and gelation properties of PFs. Cross-linking of amylopectin molecules strengthens the starchy gel network during manufacturing of potato-based snacks resulting in a crisp with a significantly lower fat content after deep-frying.



Stijn Reyniers was born on July 29 1992 in Duffel, Belgium. In 2013, he obtained a Bachelor's degree at the faculty of Bioscience engineering at the KU Leuven and finished his Master in Food Technology in 2015 at the same faculty. He currently works as a PhD student at the Laboratory of Food Chemistry and Biochemistry (KU Leuven), under the supervision of Prof. Jan A. Delcour. His research topic comprises the functionality and modification of pregelatinized starches from different botanical origins.

4.2. **Ruud van der Sman**, Wageningen (The Netherlands)
Swelling of starch microgels in sugar solutions

Detailed information will be presented at the convention.

4.3. **Koen Venema**, Maastricht (The Netherlands)
The effects of starch and starch derivatives on the gut microbiota in health and disease”

The gut microbiota is important in health and disease. It has been shown to play a role in many different diseases and disorders, ranging from colon cancer and inflammatory bowel disease, to allergy of skin and lungs, brain development and cognition, and obesity. The microbiota is a complex ecological consortium that is made up of ~250 individual-specific microbial species that interact with the host. Their major fermentative activities in the colon are the fermentation of carbohydrates and proteins. This leads to the production of microbial metabolites, which are taken up by the host. Carbohydrate fermentation leads to the, generally to be considered health-beneficial and so-called, short-chain fatty acids (SCFA; primarily acetate, propionate and butyrate), while protein fermentation leads to a range of toxic metabolites. Especially butyrate is considered a healthy metabolite, as the epithelial cells of the colon use it as an energy source, and without it the gut may get into problems and may become 'leaky'.

Microbes prefer to ferment carbohydrates over proteins, as they would like to incorporate the amino acids from proteins into their own enzyme machinery. Therefore, in the proximal colon primarily carbohydrate fermentation occurs, but in the distal colon, where the fermentable carbohydrates are depleted, this is switched to protein fermentation, with the concomitant production of toxic metabolites. Therefore, it is important to extend carbohydrate fermentation to the distal colon.

In humans it is very difficult to study fermentation in the large intestine, primarily due to the difficulties in getting access to the area. In the late 1980s therefore, the Dutch Organization for Applied Scientific Research (TNO) set out to develop a dynamic, computer-controlled in vitro model of the gastrointestinal tract (nick-named TIM). They developed a model for the stomach and small intestine (TIM-1) and one for the colon (TIM-2). These models accurately mimic the changing conditions in the GI tract as foods transit through the gut. The systems have been validated using clinical or animal data. The parameters mimicked in the in vitro model have been so accurate, that the systems can be used to predict what happens in man and animals, and can be used to study mechanistically what is going on in the gut.

The colon model, TIM-2, has been used extensively to study the effect of non-digestible carbohydrates, including resistant starch and starch-derivatives, on the composition and activity of the gut microbiota. Past experiments have shown that resistant starch (depending on which RS) may lead to the production of a high ratio of butyrate, the fuel for the colonocytes. Recent experiments show that starch is fermented more slowly than e.g. the prebiotic inulin or lactose, which would perhaps allow it to be fermented in the distal colon, thereby preventing fermentation of protein and production of toxic metabolites.

Novel sources of starch, e.g. from the waste material left after starch extraction from cassava roots, may have beneficial effects on the composition and activity of the gut microbiota. In addition, energy extraction from food components, which is one of the ways that the gut microbiota is believed to be involved in obesity, has been shown in the model to be lowest on starch. The latter experiments were based on labelling of substrates with stable isotope (^{13}C), to trace exactly what happens with these food components. This technology was set up in humans and is now tuned for clinical application.

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Prof. Dr. Koen Venema is i) founder and CEO of the company *Beneficial Microbes Consultancy*, ii) the initiator and co-organizer of the *Beneficial Microbe Conference-series*, iii) editor-in-chief of *Beneficial Microbes*, and iv) Professor at *University Maastricht - campus Venlo*, where has a chair in *Gut Microbiology*. The gut microbiota has been shown to be extremely important in health and disease. In his group the effect of food components on health and disease through the gut microbiota are studied using the sophisticated TNO in vitro models of the gastrointestinal tract (nick-named TIM).

5. Enzymes

5.1. **Bart Koops**, Leiden (The Netherlands)

A novel enzyme to improve wheat starch and gluten separation.

There are various industrial processes used for the wet-separation of wheat starch and gluten. The most common methods are the dough process, the dough batter process and the batter process. Despite the many differences in these processes, think of temperature, mixing speed, residence time etc., these processes all rely on the same principles. Two of the most important aspects for gluten starch separation are viscosity and gluten protein agglomeration. A low viscosity improves the separation of the different components, and the gluten agglomeration is important for the gluten quality.

Enzymes can improve the wheat starch-gluten separation process. Especially arabinoxylan hydrolysing enzymes, or xylanases, have been described to improve both the ease of the

process as well as the quality and quantity of starch and gluten. Such enzymes reduce viscosity by hydrolyzing the soluble arabinoxylans of wheat. In addition, xylanases can hydrolyze insoluble arabinoxylans that are thought to hinder agglomeration of the gluten protein.

To mimic the starch-gluten separation process on lab scale is challenging. In this presentation we will show different lab-scale methods that were used for the development of a new wheat starch-gluten separation product. With the combined results from the different methods we will demonstrate the benefits of this new product in the separation process.



Bart Koops is Director EMEA Biorefineries Applications at DuPont Industrial Biosciences. Bart studied biochemistry at the University of Utrecht in The Netherlands and obtained his PhD in enzymology at the same university in 1999. After working at both Royal Numico and Yakult in the Netherlands, he joined Genencor 2005, which became first Danisco and then DuPont. After being responsible for technical sales for 6 years Bart moved to his current role in 2011 as EMEA Grain Processing Applications leader. He is leading the team in developing new products and processes for the grain processing industry and providing support for customers.

5.2. **Nina Schrögel-Truxius**, Darmstadt (Germany)

Application of arabinoxylan degrading enzymes and their effects in wheat flour process”

The separation of wheat flour into starch and gluten has been understood for more than 250 years. Nowadays there are around 15 different conventional separation processes known. However, only three are predominantly in use industrially, these are called the dough, batter-dough and batter process. In the latter part of the 20th century, the introduction of enzyme preparations containing xylanases and endoglucanases has led to improvements in the separation. The addition of an enzyme preparation containing xylanase or endoglucanase activity degrades the non-starch polysaccharides, specifically arabinoxylans, of wheat, leading to a decrease in viscosity, resulting in an improved separation.

In an application-oriented laboratory scale study, the action of arabinoxylan degrading enzymes and their effects at various stages in wheat flour process was investigated. Analytical methods were developed to study the effect on the gluten quality and water stream during the separation process. Determination of gluten quality was carried out by measuring total nitrogen by the Dumas method, the molecular weight distribution of gluten proteins and the gluten coagulation index by sieve tests. Gluten extensibility was measured with a texture analyzer and a Glutopeak system and performance of the gluten assessed in baking trials. An attempt was also made to look into the effect of the enzymes on fresh water usage during the separation process.

The endoglucanase belongs to the group of nonspecific enzymes and is able to hydrolyze not only cellulose, but also xylans. It results in the highest gluten coagulation index as measured by the sieve tests. This enzyme also gave the largest extensibility as well as the greatest maximum force on gluten. The baking trials showed a slightly increase of bread volume, and the fresh water usage trials resulted in 51 % less fresh water usage compared to a control.

GH 11 xylanase, which is described by xylanase 1, is recognized for its lower catalytic specificity and acts predominantly towards WUAX (water un-extractable arabinoxylans) and gives a good solubilization of arabinoxylan. The use of xylanase 1 in the separation resulted in a gluten with the lowest coagulation index, poorer strength and greater extensibility and gave the largest increase in bread volume. This enzyme also led to the highest amount of supernatant after separation and shows a potential 51 % reduction in fresh water usage.

GH 10 xylanase, which is described by xylanase 2, is known to be predominantly active towards WEAX (water extractable arabinoxylans) with a preference towards soluble arabinoxylan. The use of the enzyme in separation gives a gluten with a good coagulation index, slightly higher strength, but a lower extensibility. In the baking trials, xylanase 2 shows just a small increase of loaf volume or even a lower volume than the control sample. This enzyme gave, with 31 %, the lowest potential amount of fresh water savings.

Analysis by SDS-gel electrophoresis showed that there is no change in the molecular weight of the gluten proteins by the application of any of the three applied enzymes.

The trials have shown that there is a correlation between arabinoxylans and the gluten network formation. For all the trials, hydrolysis of AX by enzyme addition leads to a change in gluten properties as evaluated. Furthermore, the addition of a xylanase or an endoglucanase leads to less fresh water utilization for the process, which is profitable for every customer.



Nina Schrögel-Truxius works for AB Enzymes for more than five years. In her role as Technical Service Manager for Food Enzymes she focuses on the development and application of enzyme products for the grain processing industry. She holds a Master degree in chemical engineering from Provalids University of Applied Sciences in Frankfurt, Germany.

6. Technology

6.1. **Benjamin Metz**, Vienna (Austria)

Starch sweetener plant design – practical experience

Starch hydrolysis and subsequent processing of resulting sugars allows the production of a big variety of various sweetener products. The applications for such sugars and alcohols are mainly as natural sweeteners in food and beverage industry, pharmacy or cosmetic products but also as fermentation substrate. The general design of starch processing plants strongly depends on the type of sweetener to be produced and on the necessary product quality which has to be achieved.

First criteria to distinguish starch sweeteners itself is the dextrose equivalent (DE) defining the grade of starch (DE 0) hydrolyzation to glucose (DE 100). Depending on the hydrolyzation degree the process is designed with only enzymatic / acidic conversion to dextrans (liquefaction) or further to smaller sugars (saccharification).

Low-DE sweeteners range from spray dried maltodextrin to specifically hydrolysed maltose syrups or glucose syrup with DE above 20. High-DE sweeteners, especially resulting from glucose syrups above DE 96, mainly differ due to further processing steps. Enzymatic isomerization reaction produces high fructose syrups (HFS-42, HFS-55), whereas by using catalytic hydrogenation reaction sorbitol is formed and via crystallization glucose monohydrate is produced. Quality criteria and necessary purification steps to achieve that will be discussed as well as technological options for individual process steps.

The initial plant design always needs to consider energy and water consumption to result in an overall economic process. Consequently, the re-use of secondary energy (vapor from dryers) and heat recovery from thermal processes (starch hydrolyzation, evaporation) will be discussed and examples shown with resulting total steam saving of 20%. In addition, the reduction of general water demand is a crucial factor for every project. Therefore, water consumption as well as water gains in the process will be shown and saving potentials discussed which result in reduction of water demand in the range of 60%.



Benjamin Metz works as technologist at Vogelbusch Biocommodities GmbH. 2011, he obtained his PhD in biochemistry about hemicellulose metabolism in filamentous fungi at Technical University Vienna (Austria) before he worked for two years as a post-doc at the Technical University of Delft (the Netherlands) to improve robustness of yeasts for alcohol fermentation process. Starting at Vogelbusch Biocommodities in 2013, Benjamin is specialized on designing, engineering and start-up of starch sweeteners plants.

6.2. **Laurent David**, Nancy (France)

Using modeling to compare and optimize continuous chromatographic processes

Sugar purification is one of the largest application of continuous chromatography and requires fine-tuned systems. For instance, fructose is separated from glucose to produce High Fructose Corn Syrup (HFCS 55 and 42).

These separations, typically performed on ion-exchange resins, are extremely productive: relatively linear adsorption isotherm enables very high feed concentration, up to 800 g/L. The sugar market is extremely competitive, thus fine-tuning the system to maximize recovery, productivity and minimize water consumption is of utmost importance. We have developed advanced modeling tools and simulation software to understand the key drivers for optimization.¹

In spite of their relative linearity, the glucose and fructose adsorption isotherms are slightly anti-Langmuirian (the slope increases with concentration) and synergistic (increasing fructose concentration increases glucose adsorption and vice versa).

Using the so-called equilibrium model (triangle representation) and accounting for this uncommon adsorption behaviour, we identify SMB parameters that are critical for the required separation, and we show that fructose can be purified and concentrated at the same time.

An alternative process is also investigated where fructose is purified at 90 % (instead of 55%) and a by-pass is used to obtain the targeted 55 %.

Both processes are compared in terms of equilibrium model.

Finally, using ChromWorksTM,¹ SMB, S-SMB, I-SMB and new innovative custom multi-column configurations are designed, the critical parameters are identified for each and their performances are predicted, allowing for a rational comparison of these systems.

1. ChromWorksTM, a modeling and simulation software for chromatographic separations, Ypso-Facto, Nancy, France



***Laurent David** is a process engineer specialized in biochemical and purification processes. He graduated from ENSIC (Nancy, France). During his career, Laurent has been involved in the development of a wide range of bioprocesses all over the world including organic acids, sugars, biopharmaceuticals and omega-3s. Laurent is also involved in the modeling and simulation of chromatographic and ion exchange processes.*

6.3. **Dietmar Schulze**, Wolfsburg (Germany)

Silo design for flow – Application on starch silos

Introduction: In many industrial situations, powders and bulk solids have to be stored in bins and silos. Especially at discharge often unexpected problems like flow obstructions take place. Most problems can be avoided by proper equipment design with respect to the flow properties of the bulk material to be stored.

In the presentation typical problems occurring in bins and silos are addressed. Further, a short overview on silo design procedures to avoid such problems is given, the most important powder properties to be applied on the design are outlined, and appropriate test principles are mentioned. Finally it is demonstrated how test results on a starch sample are applied on the design of a starch silo.

1 Flow problems

Most storage systems for powders and bulk solids are based on gravity flow, such as silos, hoppers, and bins, where most flow problems appear while discharging. To understand the problems, it is important to know how the material flows. Two different basic flow patterns can occur (Figure 1), called mass flow and funnel flow (also known as core flow).

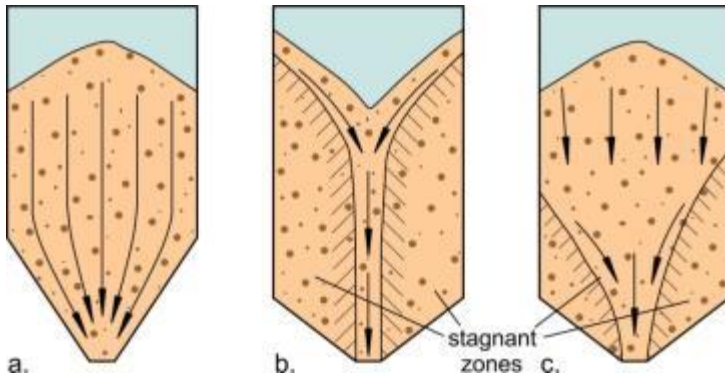


Fig. 1: Flow profiles: **a.** Mass flow; **b.** Funnel flow with stagnant zones up to the level of filling; **c.** Funnel flow with stagnant zones in the lower silo part; here the stagnant zones are asymmetric [1, 2]

In case of mass flow (Fig. 1.a), every particle of the bulk solid in the silo is moving while discharging the silo. Mass flow is only possible if the hopper walls are steep and/or low enough in friction. If the hopper wall is too flat or too frictional, funnel flow will occur. In a funnel flow silo (Fig. 1.b/c) at first only the bulk solid in a channel above the opening flows downwards. The bulk solid located in the stagnant zones, which develop at the silo periphery starting at the hopper walls directly above the opening, can be discharged only if the silo is emptied completely – provided the stagnant zones are not consolidated to such extent that they form a stable pipe (rathole), as explained later. The shape of the stagnant zones can be rather different (Fig. 1.b/c), even with the same bulk solid in the same silo. In a silo used as a buffer and never discharged completely, bulk solid can remain in the stagnant zones over long periods of time and change its properties.

Problems related to the strength of the material are shown in Figure 2 [1, 2]. If the outlet opening is too small, a stable arch can form above the outlet due to cohesive forces between the particles resulting in a certain strength of the bulk solid (Fig. 2.a). In a funnel flow silo, the bulk solid in the stagnant zones can consolidate to such an extent that it will not be able to flow out after the flow zone has emptied out. The result is a so-called rathole reaching from the outlet opening to the top of the filling (Fig. 2.b). If a bulk solid tends to increase its strength when being stored at rest (so-called time consolidation), the probability of a stable rathole is increasing.

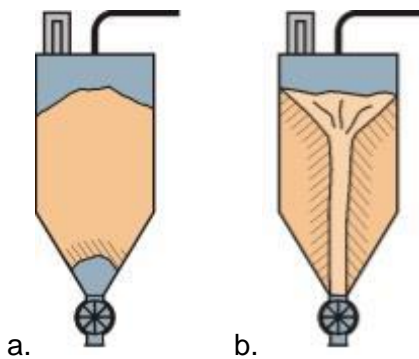


Fig. 2: Problems related to the bulk solid's strength; **a.** arching; **b.** ratholing (piping) [1, 2]

Funnel flow (Fig. 3.a) is the source of a couple of other problems that will be described in short. The residence time distribution of the bulk solid in a funnel flow silo can be rather broad. Especially the material in the lower part of the stagnant zones may remain there for a longer period of time – until the silo is emptied completely (nearly “first in – last out”). Thus, product quality (and strength, as discussed above) may change due to long residence times. On the other hand, the residence time of the bulk solid in the flow zone of a funnel flow silo can be extremely short (Fig. 3.c), so that the material, which has just been fed into the silo, is immediately discharged. Within this short time an easily fluidized bulk solid (e.g., flour, fine chalk) cannot sufficiently deaerate. Hence, it will flood out of the outlet opening like a fluid, resulting in increased dust generation and flooding of the feeder [1, 2].

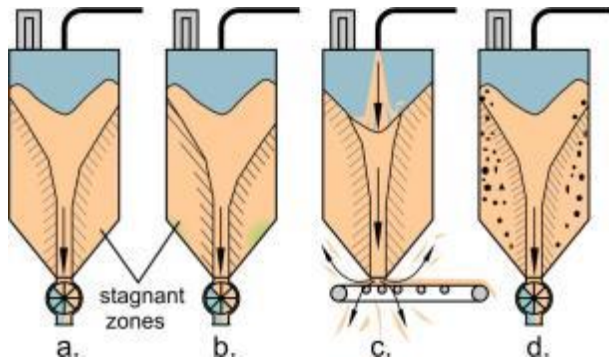


Fig. 3: Operation of funnel flow silos and possible problems; **a.** funnel flow; **b.** funnel flow with wide residence time distribution and deterioration in product quality; **c.** flooding; **d.** segregation [1, 2]

Funnel flow can also result in reduced product quality due to segregation (Fig. 3.d). When filling a silo, one has to take into account that the product can segregate across the cross-section of the silo (e.g., more fines in the center, more coarse particles in the periphery). If this happens in a funnel flow silo, at first the material from the center (the fines) is discharged, followed by the coarser material from the silo periphery.

Most of the problems discussed above can be avoided if a silo is designed for mass flow: Stagnant zones do not occur in mass flow, the residence time distribution is very narrow, and segregation over the cross-section due to the filling process is reduced since material from the center and the periphery is reunited in the hopper. Only the problem of arching remains which can be solved by a sufficiently large outlet opening.

Therefore, to design a silo for mass flow and unobstructed flow, two steps are necessary: First, the determination of the steepness of the hopper walls to avoid stagnant zones, and second, the calculation of the outlet souse to avoid arching. Both steps can only be performed if the relevant properties of the bulk solid (flow properties) are known. The most important quantity for the determination of the hopper slope is the **wall friction angle**, which is the friction angle between the bulk solid and the surface of the hopper wall. The major quantity regarding outlet dimensions is the **strength** of the consolidated bulk solid.

2 Flow properties: The yield locus concept

Around 1960 Andrew W. Jenike worked on a theory to describe the flow of bulk solids in silos [3, 4]. Jenike defined the so-called yield locus (a yield limit of a consolidated bulk solid) as a means to describe powder properties, developed an approach for hopper design (mass flow, no arching, no ratholing), and introduced an appropriate test method (shear test).

Here only a brief introduction to Jenike's approach can be presented. For more detailed information the reader is referred to Jenike's original papers [3, 4], or other references where the theory is described, e.g. [1, 2, 5, 6].

To understand the yield locus concept it is helpful to know the relationship between strength and stress, or, more detailed, between the unconfined yield strength (compressive strength) and the consolidation stress. Figure 4 shows a hollow cylinder filled with a fine-grained bulk solid (cross-sectional area A ; internal wall of the hollow cylinder assumed as frictionless). The bulk solid is consolidated by the stress σ_1 – the consolidation stress – in the vertical direction. This results in an increase of the bulk density which goes along with an increase in strength of the bulk solid specimen.

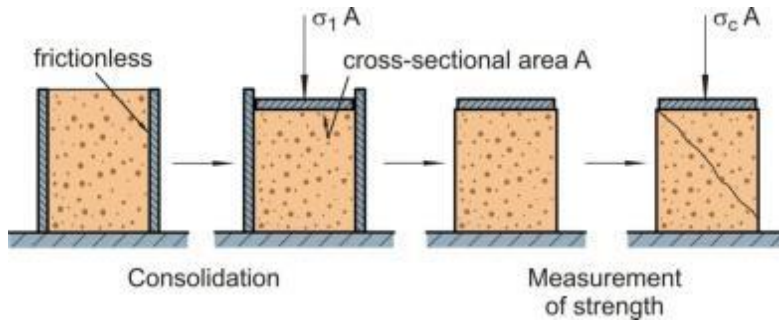


Fig. 4: Uniaxial compression test [1, 2, 7]

After consolidation, the bulk solid specimen is relieved of the consolidation stress, σ_1 , and the hollow cylinder is removed. If subsequently the consolidated cylindrical bulk solid specimen is loaded with an increasing vertical compressive stress, the specimen will break (fail) at a certain stress. The stress causing failure is called compressive strength or unconfined yield strength, σ_c .

Uniaxial compression tests (Fig.4) conducted at different consolidation stresses, σ_1 , lead to different values of bulk density, ρ_b , and unconfined yield strength, σ_c . Plotting unconfined yield strength, σ_c , over consolidation stress, σ_1 , and drawing a curve through these points usually results in curves like those for product A in Fig. 5 where unconfined yield strength, σ_c , typically increases with consolidation stress, σ_1 . The curve $\sigma_c(\sigma_1)$ is called the flow function.

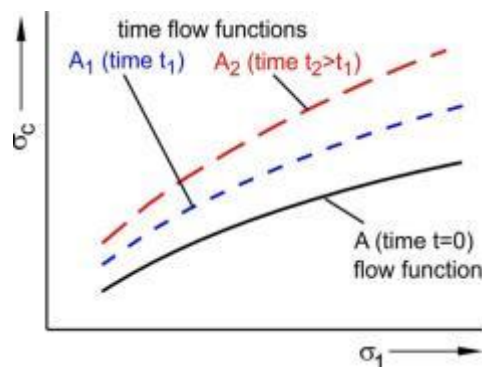


Fig. 5: Instantaneous flow function and time flow functions for two different storage times t_1 and $t_2 > t_1$ [1, 2, 7]

Some bulk solids increase in strength if they are stored for a longer time at rest while subjected to a compressive stress. This effect is called time consolidation. Typical causes of time consolidation are, e.g., chemical processes, crystallization between the particles, enlargement of the contact areas through plastic deformation or capillary condensation [1, 2].

With the test shown in Fig. 4 long-term storage in a silo can be simulated by loading the specimen with the consolidation stress, σ_1 , for a defined period of time, t_1 , e.g., one day, or one week. Then the unconfined yield strength is determined following the principle explained above (Fig. 4). If a bulk solid exhibits time consolidation, one obtains curves $\sigma_c(\sigma_1)$ for storage periods $t > 0$ (curves A_1 , A_2 in Fig. 5) which are called time flow functions.

The uniaxial compression test (Fig. 4) is fine to explain the strength-stress-relationship, but it is rather problematic for testing fine-grained, cohesive bulk solids, because one obtains unconfined yield strength values that are too low [9], and preparation of the hollow cylinder to obtain frictionless walls is very time-consuming. In addition, further important parameters (e.g., internal friction and wall friction) cannot be determined with this test.

In advanced bulk solids technology shear testers are used to measure yield loci of bulk solids (as proposed by Jenike [3, 4]). For a shear test, a bulk solid specimen is loaded vertically by a normal stress, σ (Fig. 6.a). Then a shear deformation is applied on the specimen by moving the top platen with a constant velocity, v . This results in a horizontal shear stress, τ , originated by the friction between the particles (Fig. 6.b).

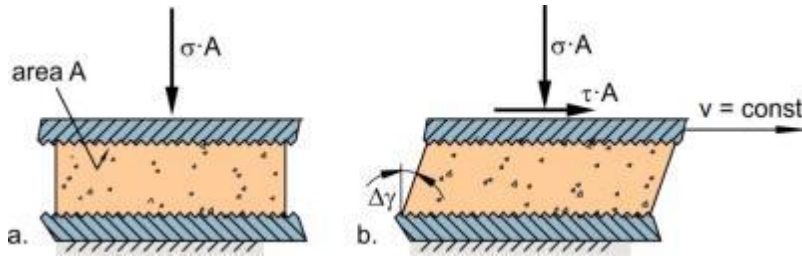


Fig. 6: Specimen in the shear cell of a shear tester: **a.** initial loading with normal stress σ ; **b.** shear deformation (velocity $v = \text{const}$) [1, 2]

When a point of a yield locus is measured, two steps are necessary in analogy to the uniaxial compression test: First the bulk solid specimen is consolidated. This is called “preshear”. Subsequently a point of the yield locus is measured. This step is called “shear” or “shear to failure”. A detailed description of the procedure is given in [1, 2, 4, 10].

Based on such tests, for each consolidation stress a yield locus can be measured. An example is shown in Fig. 7. The yield locus is the yield limit of a consolidated bulk solid. From the yield locus the parameters representing the flow properties are determined. Consolidation stress, σ_1 , and unconfined yield strength, σ_c , result from Mohr stress circles drawn on the diagram [1 – 4]. With consolidation stress, σ_1 , and unconfined yield strength, σ_c , one can calculate flowability, ff_c :

$$ff_c = \sigma_1 / \sigma_c \quad (1)$$

The larger ff_c is, i.e., the smaller the ratio of the unconfined yield strength to the consolidation stress, the better a bulk solid flows. Similar to the classification used by Jenike [3, 4], one can define flow behavior based on ff_c as follows:

$ff_c < 1$	not flowing
$1 < ff_c < 2$	very cohesive
$2 < ff_c < 4$	cohesive
$4 < ff_c < 10$	easy-flowing
$10 < ff_c$	free-flowing

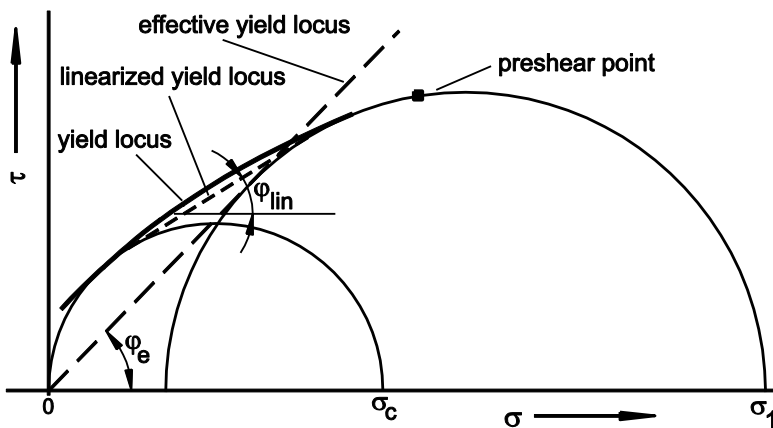


Fig. 7: Yield locus and parameters [1, 2, 7]

Flowability, ff_c , allows for a comparison of different bulk solid samples, e.g., a bulk solid mixed with different amounts of a flow agent, or produced in different ways. The advantage of this method is that it is based on a well-defined physical quantity, namely the unconfined yield strength. ff_c is often applied on comparative tests or product optimization.

A straight line through the origin of the σ, τ -diagram, tangent to the greater Mohr circle (representing the stresses at the end of consolidation), is the effective yield locus as defined by Jenike [3, 4] (broken line in Fig. 7). φ_e can be regarded as a measure of the internal friction in the flowing bulk solid and is required for silo design for flow according to Jenike's theory.

If several yield loci are measured at different stress levels, each yield locus represents another state of consolidation and, thus, another bulk density and another effective angle of internal friction. These flow properties can be plotted as a function of the consolidation stress, σ_1 , similar to Fig. 5 where unconfined yield strength is plotted vs. consolidation stress. Time consolidation, which describes the increase of the unconfined yield strength with time during storage at rest, is measured with a shear tester similar to the measurement of a yield locus [1 – 4]. The difference is that the specimen (after being consolidated by the preshear step, but before shear to failure) is subjected to a static load for the selected consolidation period.

The most important quantity for the determination of the hopper wall slope to achieve mass flow is the friction between the bulk solid and the wall of the hopper. It is characterized by the wall friction angle. The principle of a wall friction test is shown in Fig. 8. The bulk solid specimen is subjected to a vertical normal stress. The normal stress acting between bulk solid specimen and wall material is called the wall normal stress, σ_w . The bulk solid specimen is then shifted relative to the wall material surface with a constant velocity, v . The shear stress acting between bulk solid specimen and wall material is measured. This is done at different levels of normal stress. All pairs of values of wall normal stress and wall shear stress are plotted in a σ_w, τ_w -diagram (Fig. 9). The curve (or line) running through the measured points is called the wall yield locus.

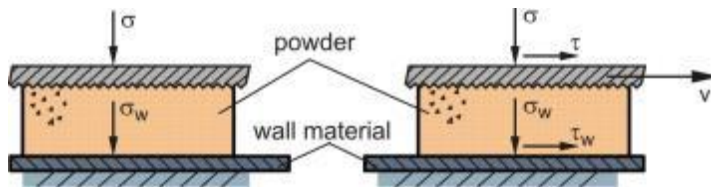


Fig. 8: Principle of a wall friction test [1, 2]

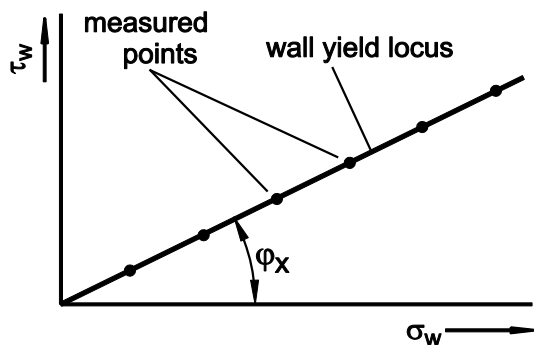


Fig. 9: Wall yield locus [1, 2]

The wall yield locus is a yield limit like the yield locus. The wall yield locus describes the wall shear stress, τ_w , necessary to shift a bulk solid continuously across a wall surface under a certain wall normal stress, σ_w . To quantify wall friction, the wall friction angle, ϕ_x , is used. It is the slope of a line running through the origin of the σ_w, τ_w -diagram and a point of the wall yield locus. If the wall yield locus is a straight line running through the origin, the wall friction angle is the slope angle of the wall yield locus as in Fig. 9.

3 Shear testers

Jenike [3, 4] introduced the Jenike Shear Tester (Fig. 10.a) which works according to the principles shown in Figs. 6 and 8 where the bulk solid specimen is subjected to a translational shear deformation. The specimen is contained in a shear cell consisting of a base of circular cross-section, a shear ring of the same diameter lying above the base, and a shear lid. The shear lid is loaded centrally with a normal force, F_N . To shear the specimen, the upper part of the shear cell is moved horizontally. Force F_S required to do this is measured. For the measurement of wall friction, the base of the shear cell is replaced by a sample of wall material (Fig. 10.b). The test procedure for the Jenike shear tester is described in detail in [1, 2, 10, 11]

Since the operation of the Jenike tester is difficult and time-consuming, it was rarely applied on other tasks than silo design. Progress was made with the development of rotational shear testers, e.g., the first ring shear tester for powders and bulk solids [12] where the relative displacement is achieved by rotation of the top of the annular powder specimen relative to the bottom.

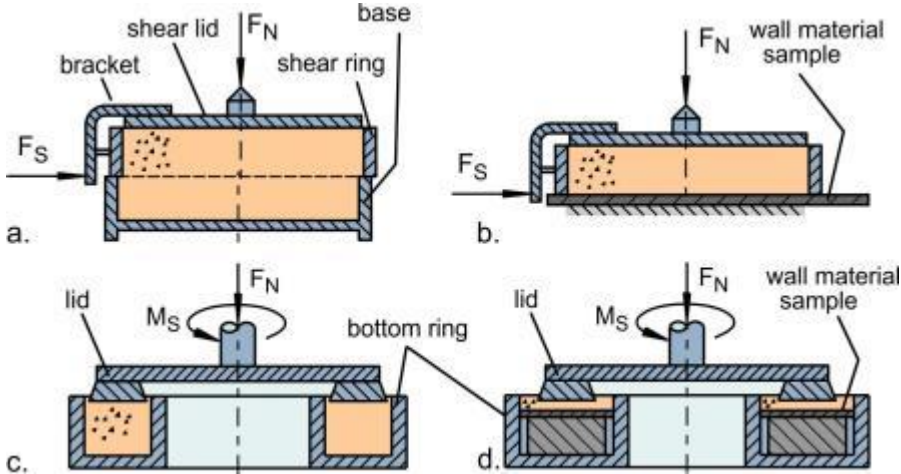


Fig. 10: Shear cells with powder specimens (schematic); **a.** Jenike shear tester, **b.** Set-up of Jenike shear cell for wall friction tests, **c.** Ring shear tester, **d.** Ring shear cell for wall friction tests [7, 8]

The specimen in the shear cell of a ring shear tester (Figs. 10.c/d) is loaded vertically with a normal force, F_N , by the annular lid. By rotating the bottom ring relative to the lid around the vertical axis of the shear cell, the specimen is subjected to shear deformation. Shear stress is calculated from moment, M_S , acting on the lid. For the measurement of wall friction with a ring shear tester, a bottom ring containing a sample of the wall material under consideration is used (Fig. 10.d).

Ring shear testers were developed already in the 1960's, but these devices were relatively heavy and did not allow all tests and conditions as known from the Jenike tester. In 1992/93 the author has developed a new principle of ring shear tester [8] which is lighter and provides all tests offered by the Jenike tester, but less time-consuming and more accurate. As a result of these developments, ring shear testing has become more and more popular, not only for silo design, but also for product development or quality control.

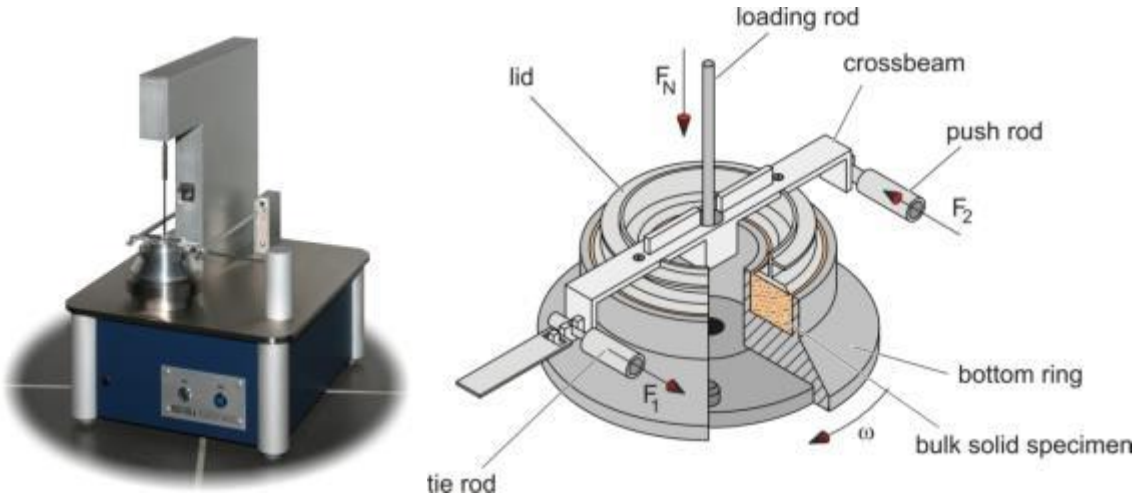


Fig. 11: Ring Shear Tester RST-XS.s and shear cell [1, 2, 8, 13]

Figure 11 shows the ring shear tester type RST-XS.s which was used to measure flow properties of starch reported in the present paper, and a shear cell. The specimen is located in the rotating annular bottom ring and subjected to a vertical stress by a loading rod exerting the normal force F_N on the lid. The shear stress is determined from forces, F_1 and F_2 , acting in the tie rod and push rod, both preventing the lid to rotate. For wall friction tests a similar shear cell is used where the wall material sample is forming the bottom of the bottom ring, and the bulk solid specimen is located on top of the wall material sample (Fig. 10.d).

4 Application of shear test results on silo design

The design of a silo for the storage of starch is demonstrated in the following. Starch is a fine-grained, cohesive bulk solid with a significant tendency to time consolidation. Wall friction is strongly dependent on the wall material.

For the presentation tests on different samples of starch have been performed. Silo design is done for one type of maize starch. For demonstration purposes time consolidation test results are added which are taken from former project reports [14] and slightly changed to keep confidentiality. It has to be noted that different starch types probably result in different flow properties and, thus, different silo design results. Thus, the results published here cannot be applied on other starch silo projects.

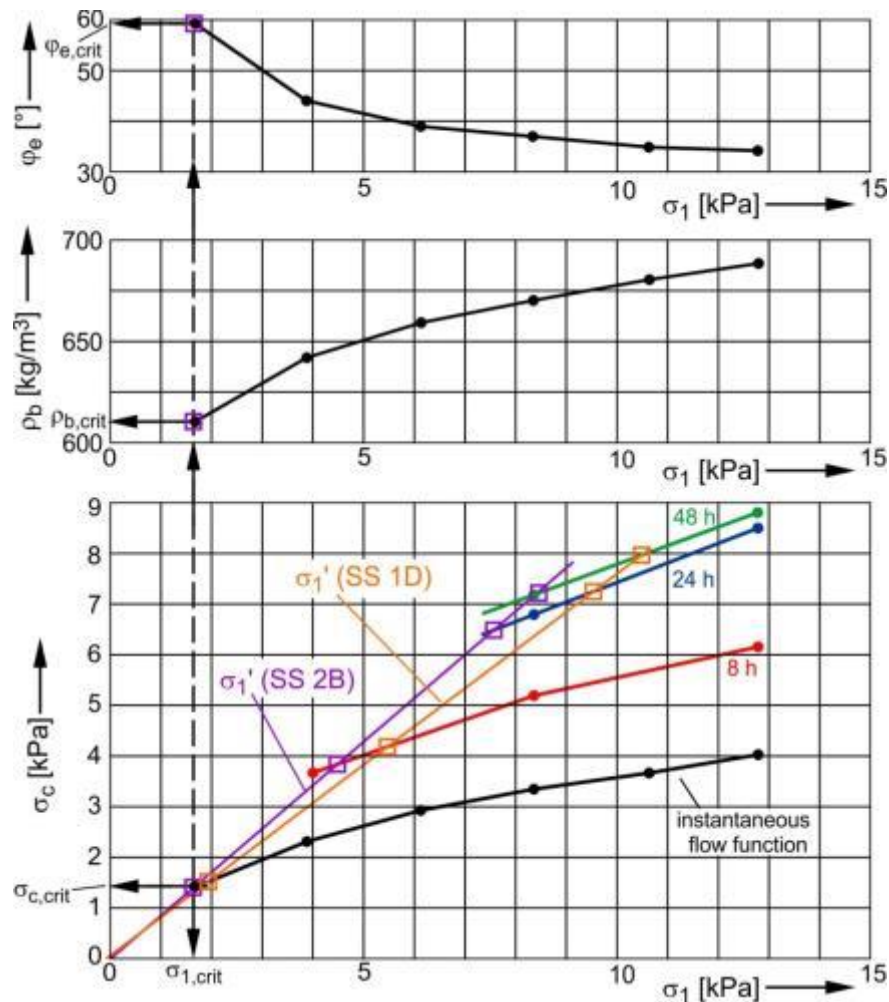


Fig. 12: Flow properties of a sample of maize starch (circles represent test points resulting from yield locus tests); results of time consolidation tests taken qualitatively from former tests on various maize starch samples [14]; lines σ_1' represent the major stress in an arch; dashed lines, arrows and squares indicate the silo design procedure

The flow function (as explained in Section 2, Fig. 5) was measured for consolidation stresses, σ_1 , of 1.7 to about 13 kPa (Fig. 12; each point represents the result of one particular yield locus test). Further, time consolidation results have been added forming three time flow functions. It is clearly visible that the unconfined yield strength, σ_c , increases significantly with time (e.g. at consolidation stress, σ_1 , of about 8.4 kPa), but most of the increase happens within 24 hours:

The increase of strength from 24 hours to 48 hours of storage at rest is relatively small, and the same was found for even longer storage periods.

The upper diagrams in Fig. 12 represent the bulk density, ρ_b , and the effective angle of internal friction, φ_e , characterizing the internal friction of the bulk solid. Wall friction was measured for two different wall materials, namely cold-rolled stainless steel (2B finish, DIN EN 10088-2) and warm-rolled stainless steel (1D, DIN EN 10088-2). On the diagram of Fig. 13 the wall friction angles are plotted over wall normal stress, σ_w . The rougher surface of the warm-rolled stainless steel results in significantly higher wall friction angles. Towards lower stresses the wall friction angles are increasing. This is probably a result of adhesive forces acting between wall surface and bulk solid.

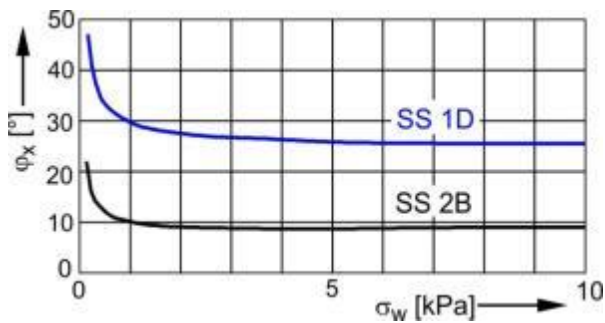


Fig. 13: Wall friction angles of the starch sample against warm-rolled (SS 1D) and cold-rolled (SS 2B) stainless steel

Jenike's approach for silo design concentrates on conical and wedge-shaped hoppers (Fig. 14). Also asymmetric wedge-shaped hoppers are treated and instructions are given for asymmetric conical hoppers, but, however, these asymmetric shapes are disadvantageous considering discharge behavior or best use of space.

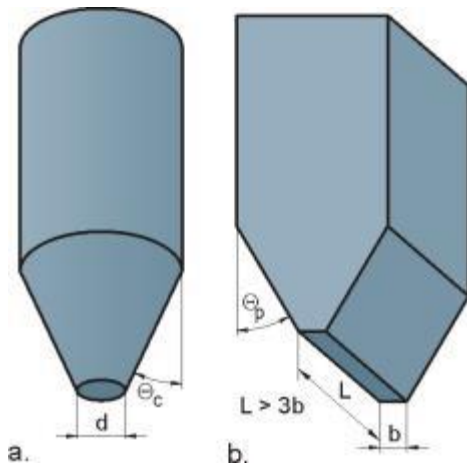


Fig. 14: Basic silo shapes [3, 4]: **a.** conical; **b.** wedge-shaped

Jenike's approach [3, 4] allows determining the maximum hopper wall slope to achieve mass flow. Boundaries between mass flow and funnel flow for conical hoppers are plotted in Fig. 15. The boundaries, which depend on the internal friction represented by the effective angle of internal friction, φ_e , give the maximum theoretical hopper wall slope, Θ_c , for a certain wall friction angle, φ_x . For the two wall materials tested (Fig. 13) the wall friction angles found at a particular wall normal stress are plotted on the mass flow diagram in Fig. 15 (explanation of stress selection follows). The slope angles, Θ_c , found at the boundary are reduced by a safety margin of 3° (see arrows in Fig. 15) resulting on a maximum hopper wall slope of 35° for cold-rolled stainless steel (SS 2B), and only 13° for the more frictional warm-rolled stainless steel (SS 1D). Because of the shallower wall inclination, it was decided to use cold-rolled stainless steel as lining of the hopper walls. To gain some additional safety, e.g., to account for smaller changes of the bulk solid's behavior, the slope of the hopper walls was chosen to be 30° .

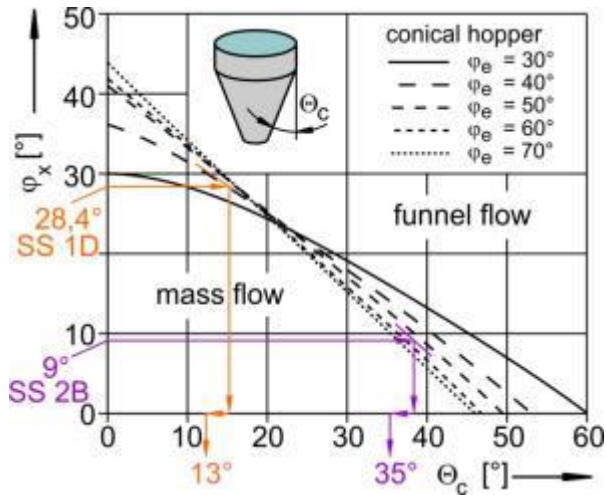


Fig. 15: Mass flow diagram (conical hopper) and determination of maximum hopper slope for measured wall friction angles [1, 2]

The next step is the determination of the critical outlet size. The principle is shown in Fig. 16. In the lower part of a mass flow hopper the major stress (consolidation stress), σ_1 , is proportional to the local hopper diameter. Thus, it is equal to zero at the virtual hopper apex and increases in the upward direction. Knowing σ_1 , and knowing the flow function (Fig. 12), the unconfined yield strength of the bulk solid, σ_c , can be plotted over the height of the hopper, too.

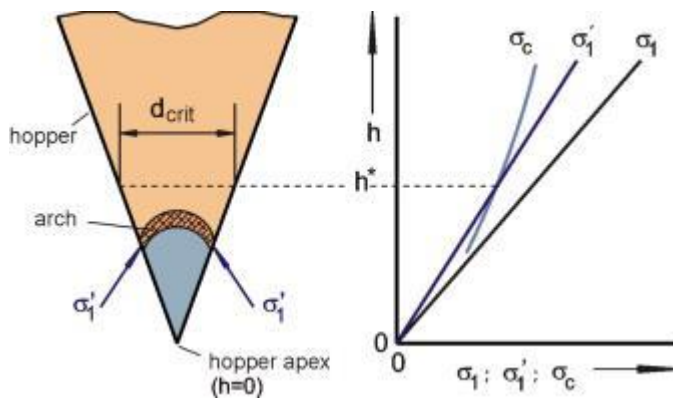


Fig. 16: Determination of minimum outlet size, d_{crit} , to avoid arching [1, 2]

If a cohesive arch has formed in a hopper (Fig. 16), a force resulting from the weight of the bulk solid is transferred to the hopper walls. This effect is represented by the major stress required to support a stable bulk solid arch, σ_1' . σ_1' can be calculated and is proportional to the local hopper diameter (Fig. 16) [3, 4].

A stable arch is only possible in that part of the hopper where the unconfined yield strength is greater than the stress that would exist in a stable arch ($\sigma_c > \sigma_1'$), i.e., beneath the point of intersection of the σ_c curve with the σ_1' line (Fig. 16). The intersection point defines that position in the hopper (height h^* , Fig. 13.21) where the hopper diameter is equal to the so-called critical outlet diameter, d_{crit} , which must be exceeded if arching is to be avoided.

If a bulk solid has the tendency to consolidate with time when stored at rest, in analogy to the (instantaneous) flow function, time flow functions exist which represent the unconfined yield strength after certain consolidation periods (refer to time flow function in Fig. 12). If the time flow functions would be transferred into Fig. 16, a point of intersection of σ_1' and σ_c would result located further upwards, i.e., at $h > h^*$. Thus, a larger critical diameter, d_{crit} , would result. This leads to the well known effect that with increasing time of storage at rest larger outlet diameters are necessary to avoid arching.

For the practical determination of critical outlet dimensions Jenike proposed a procedure where stress σ_1' is plotted on the flow function diagram as already done in Fig. 12 for both wall

materials (conical hopper). The slopes of the σ_1' lines are determined from bulk solid properties (φ_x, φ_e) and the hopper shape [1 – 4].

The point of intersection of the major stress in the arch, σ_1' , and the flow function in the σ_c, σ_1 diagram (Fig. 12) is equivalent to the point of intersection in Fig. 16. Thus, the consolidation stress, σ_1 , at this point represents the consolidation stress in the hopper where the diameter is equal to the minimum outlet diameter, d_{crit} . Therefore, coordinates of the intersection point are provided with index “crit” (unconfined yield strength, $\sigma_{c,crit}$ and consolidation stress, $\sigma_{1,crit}$). Knowing critical consolidation stress, $\sigma_{1,crit}$, bulk density, $\rho_{b,crit}$, and effective angle of internal friction, $\varphi_{e,crit}$, can be determined from the functions plotted on the diagrams of Fig. 12.

Finally the equivalent local hopper diameter, the critical diameter, d_{crit} , has to be calculated with the following equation (for details refer to [1 – 4]):

$$d_{crit} = H(\Theta_c) \frac{\sigma_{c,crit}}{g \rho_{b,crit}} \quad (2)$$

Here the critical values following from the diagrams in Fig. 12 have to be used. Function $H(\Theta_c)$ takes into account hopper geometry and hopper wall inclination) [1 – 4], g represents the acceleration due to gravity.

In a similar way the outlet dimensions can be determined which are required to avoid arching after longer periods of storage at rest (= no bulk solid is discharged). For this case the point of intersection of the major stress in the arch and the time flow function for the storage time in question, $\sigma_c(\sigma_1, t)$, has to be determined (Fig. 12), resulting in corresponding values of the critical flow properties used in Eq. (2). The calculated outlet dimensions of a mass flow hopper lined with cold-rolled stainless steel are depicted in Table 1.

Storage time at rest, t [h]	d_{crit} [m]
0	0.7
8	1.6
24	2.7
48	2.9

Table 1: Critical outlet dimensions (rounded) for the tested starch sample (conical hopper, walls lined with cold-rolled stainless steel, wall inclination $\Theta_c = 30^\circ$)

The design procedure can be more complex than it may appear following the above brief description. Especially if the effective angle of internal friction and/or the wall friction angle vary with stress as in the case under consideration, these parameters have to be estimated first, and to be corrected later by an iterative process [1 – 4]. Such iteration has been applied here and finally resulted in the wall normal stress for which the wall friction angles applied in Fig. 15 were determined.

The results of the design process according to Table 1 allow the comparison of different silo concepts. As explained earlier, the slope of the hopper walls, Θ_c , was chosen to be 30° to the vertical, and hopper walls have to be lined with sheets of cold-rolled stainless steel. The silo shown in Fig. 17.a is designed to avoid arching for a maximum storage time of 48 hours (since strength does not seem to increase much more during longer storage periods, it may be sufficient for even longer storage times, but this had to be verified by further time consolidation tests). Advantages of the large outlet opening are that the bulk solid will flow out alone due to gravity after a storage time at rest of 48 hours, and best use of space. However, the solution requires a large feeder, e.g., a vibrating hopper (live bottom, vibrating bin discharger).

The silo shown in Fig. 17.c is provided with an outlet of only 0.7 m diameter which is sufficient only for storage time zero. This means that even after a short standstill the bulk solid may form a stable arch. Thus, in the region above the outlet up to diameter 3 m discharge aids (e.g., air injection systems, air cannons) are required to initiate flow. Once flow has started, further

action of the discharge aids is not necessary since the time consolidation effect diminishes due to the movement of the particles relative to each other. The advantage of this concept is the smaller feeder, but disadvantages are the required installation of discharge aids which have to be operated in an appropriate way, the more irregular flow due to the action of the discharge aids, and the more complicated operation of the silo.

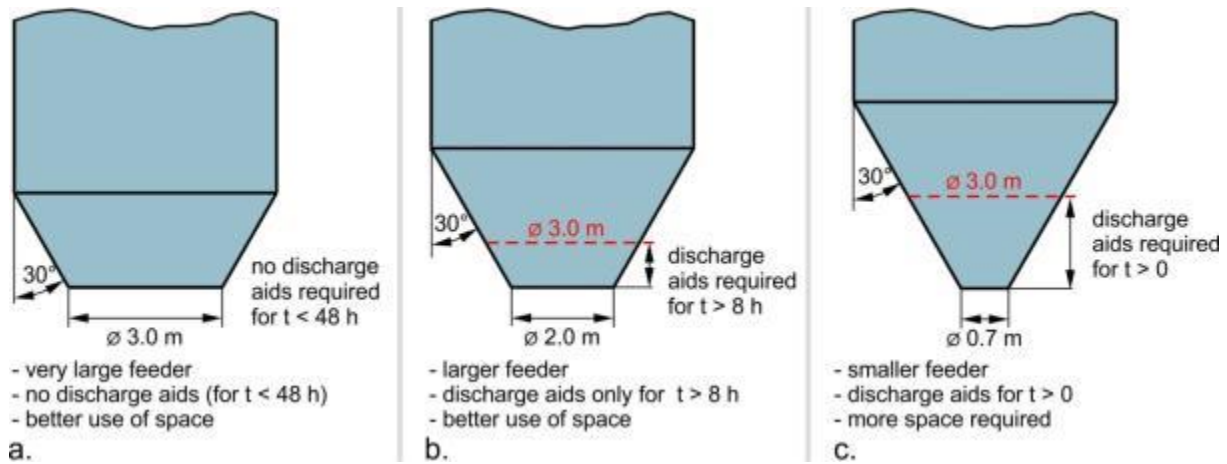


Fig. 17: Silo concepts (starch silo)

A compromise might be the concept shown in Fig. 17.b. The outlet is smaller than required for 2 days storage at rest, but a bit larger than that required for 8 hours. Thus, the outlet is sufficiently large to avoid arching after short standstills of up to about 8 hours (probably a bit more, see Table 1). To initiate flow after longer standstills, for the region between the outlet and diameter 3 m discharge aids have to be installed. In contrast to the concept of Fig. 17.c, the discharge aids have to be operated only after longer standstills in order to initiate flow.

Summary

Flow obstructions and other problems like flooding or segregation can be avoided if silos, bins, and hoppers are designed for mass flow. To achieve mass flow, the hopper walls must be sufficiently steep. The required wall slope depends strongly on the wall friction angle and to a certain extent on the internal friction of the bulk solid.

In addition to a sufficient steepness, the outlet size must be large enough to avoid the formation of stable arches. The required dimensions depend on the unconfined yield strength of the bulk solid. If the strength of the bulk solid increases with time (time consolidation), this has to be taken into account.

Silo design is only possible if the flow properties (unconfined yield strength including time consolidation, bulk density, wall friction, internal friction) are known. Thus, these quantities have to be measured with appropriate shear testers. The latter was done on samples of starch, and finally the application of these quantities on silo design was demonstrated.

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Prof. Dr. Dietmar Schulze is Professor of Mechanical Process Engineering at the Ostfalia University of Applied Sciences in Wolfsburg/Germany since March 1996. He is an expert in the areas of powder characterization and handling, partner in the consultancy “Schwedes + Schulze Schüttguttechnik GmbH”, and owner of the company “Dr. Dietmar Schulze Schüttgutmesstechnik” manufacturing ring shear testers for powder testing.

6.4. **Dominik Wüst**, Stuttgart (Germany)

Hydrothermal conversion of starchy residues – Platform chemicals and char from old bakery goods

Das Deutsche Bäckerhandwerk produziert und verkauft 4.943.400 Tonnen frisches Brot und Brötchen im Jahr 2016. Es gibt bisher keine Studie, die die Menge an verworfenen Backwaren bundesweit verlässlich abbildet, aber nach Angabe verschiedener Literaturstellen liegt diese aus Einzelhandel und Produktion bei ca. 15-20 %. Für Deutschland ergibt sich überschlagen eine Menge von maximal 682.200 Tonnen und dementsprechend von ca. 341.100 Tonnen (ca. 50 %) Kohlenhydraten. Die Verwertung von Altbackwaren seitens der Hersteller erfolgt zu ca. 17 % als Biogas und 20 % als Tierfutter mit abnehmender Tendenz. Altbackwaren bzw. die darin enthaltenen Kohlenhydrate sind jedoch ein sehr attraktives Ausgangssubstrat für die hydrothermale Umwandlung zu Plattformchemikalien, wie bspw. 5-Hydroxymethylfurfural (5-HMF), wobei ein kohleartiges Material als Nebenprodukt anfällt. Nach aktuellem Forschungsstand, einer Simulation des Prozesses mit der Software AspenPlus® und unter Berücksichtigung der aktuellen Verwertung in Deutschland besteht die Möglichkeit rund 28.000 Tonnen HMF aus Altbackwaren zu produzieren. In dieser Größenordnung ist eine Weiterverwertung zu vergleichsweise hochpreisigen bio-basierten Spezialpolymeren das ökonomisch Sinnvollste. Die als Nebenprodukt produzierte Biokohle könnte einerseits mit einem attraktiven Brennwert von 21,8 J g⁻¹ im Bereich von Braunkohle aber andererseits mit einem vielversprechendem Kohlenstoff-zu-Stickstoff-Verhältnis von 22,5 z.B. in landwirtschaftlich intensiv genutzte Böden untergepflügt werden, um diese zu regenerieren. Eine wichtige Markteintrittsbarriere liegt in der fehlenden Demonstration der Technologie im Rahmen einer Pilotanlage begründet. Hierfür konnte die AVA Green Chemistry Development GmbH aus Deutschland, die die Hydrothermale Konversion bereits im industriellen Maßstab betreibt, für die Machbarkeitsphase als Konsortialpartner gewonnen werden.



Mr. Dominik Wuest is chief engineer at the chair of Conversion Technologies of Biobased Resources. After finishing the studies of Environmental Engineering at the University of applied Sciences at Weihenstephan-Triesdorf he has worked on Hydrothermal Carbonization (HTC). Currently, he works to optimize established Renewable Energies and composting methods regarding carbon and energy efficiency as well as their ecological footprint. Furthermore, his subject is advancing in research and development of thermochemical processes including recovery of nutrients and platform-chemicals. The latter Mr. Wuest is deepening in his PhD thesis on the topic “Exploitation of platform chemicals from vegetable waste material – Chicory Roots as sources for the chemical industry”. The background of all efforts is creating bio-refinery concepts.

14th European Bioethanol and Bioconversion Technology Meeting

1. Legal Aspects

- 1.1. **Stefan Walter**, Berlin (Germany)
New legal framework under a revised Renewable Energy Directive for bioethanol from cereals

In November 2016 the EU-Commission presented its so called Winter Package, a new package of measures with the goal to achieve “Clean Energy for all Europeans”. The package includes 8 different legislative proposals covering for example energy efficiency, energy performance in Buildings, a single European market for energy etc. Three main goals are pursued by the EU-Commission:

- improving energy efficiency
- demonstrating global leadership in renewable energies
- providing a fair deal for consumers

The main part of the package is an initial proposal of the recast of the Directive 2009/28/EC (RED) with the provision of a new legal framework which is needed to facilitate the clean energy transition of Europe. The Commission suggests a binding 27%-renewable energies share in the EU in 2030 and several new rules and subtargets for renewable energies (RES) in the transport sector, e.g. for advanced alternative biofuels. Fuel suppliers should be encouraged through a binding mandate for advanced biofuels. Food and feed-based conventional biofuels, e.g. from crops or sugar, should be reduced and gradually phased out till 2030. The proposal is actually being discussed between the EU-Commission, the European Parliament (EP) and the Council of the European Union.

As EP and EU-Council did not agree on the propositions of the Commission, so called trilogue-negotiations started in spring 2018 to reach an agreement between Council and EP. The Council for example proposed an obligation ensuring an achievement of 14% RES in the transport sector including crop-based biofuels and a cap of 7 % for conventional biofuels while giving member states the possibility to lower the cap. The EP in turn suggested a 12% target for RES in transport including conventional biofuels and national caps for conventional biofuels. Negotiations are expected to be completed before summer 2018.



Stefan Walter, Lawyer and Managing Director of the German Bioethanol Industry Association (BDBe) in Berlin. Born in July 1974 in Celle, studies of law at the Leibniz-University Hanover (1993 – 1999) and EU-law at the Katholieke Universiteit Leuven (1999 – 2000). He has a few years working experience as policy advisor in different associations in Berlin. From 2014 to 2018 senior policy advisor tax and head of legal department at the German Farmers Association (DBV).

2. Raw material

- 2.1. **Arjen van Tuijl**, Leiden (The Netherlands)
Processing beyond the limits of wheat ethanol manufacturing

Compared to corn processing plants, wheat processing plants have a lower dry solids% limitation due to high viscosity of the mash. This high viscosity is mainly caused by soluble pentosans like Arabinoxylan, resulting in higher energy need for pumping, and viscosity related problems in mixing, cooling and heat-exchangers. Ultimately, this limits the throughput – thus profit – of wheat processing plant.

With DuPont's New Viscosity Reducing Enzyme, we introduce a product with outstanding viscosity reduction in Wheat Dry Grind Ethanol Production. This New Viscosity Reducing

Enzyme makes it possible to run at much higher Dry Solids (DS%), while keeping viscosity the same or even lower.

Approximately 500 cP is often the maximum viscosity which pumps can handle. This translates to a maximum DS% of around 27-28% without viscosity reducing enzyme. With current viscosity reducing enzymes this can be increased to 31-32 %DS. However, with the New Viscosity Reducing Enzyme dry solids can be increased up to - or even above - 36 %DS.

Benefits of increased dry solids not only comprise a higher throughput (more ethanol produced / hr) but also a higher yield of ethanol per DS%, as we have demonstrated in lab trials. Optimum yield was obtained in the range 34-36 %DS.

Using DuPont's New Viscosity Reducing Enzyme, wheat processing plants can be operated at much higher DS%, thus approaching corn plants with regards to output.

Summarizing, DuPont's New Viscosity Reducing Enzyme delivers for a 100MLPY wheat plant:

- 2-4% dry solids increase in liquefaction and fermentation
- Resulting in 6-13% increased solids throughput (base line 32 %DS)
- Energy saving of up to 15%
- Significant reduction of fixed cost per unit of ethanol
- Significant reduction of water consumption of up to 17%
- Reduced GHG emissions and improved sustainability

3. Enzymes

3.1. **Nadia Ramirez Angulo**, Leiden (The Netherlands) Enzymatic tools for improvements in thin-stillage evaporation

Several customers in the EMEA fuel ethanol industry have indicated they would benefit from any improvement in the downstream processing, where viscosity is one the biggest issues for the industry. Lower viscosity will facilitate solid liquid separation and evaporation of thin-stillage which is cheaper than drying.

Several components, both soluble and insoluble, are present in stillage. Soluble components like fibers can give high viscosity but also the insoluble material binds significant amounts of water, making solid / liquid separations and evaporation difficult. In this presentation, we will focus on the reduction of viscosity in evaporation of thin-stillage.

Industrial syrup samples from corn and wheat ethanol plants were analyzed to find out which enzymes have the biggest effect on viscosity reduction.

Once we know which enzymes show the best performance, the key question is where to add these enzymes in the process to maximize benefits.

Enzyme addition can be done in evaporation, but due to high temperature and relative short residence time, the required enzyme dose will be high. Therefore, fermentation is the preferred point for enzyme addition, because of the low temperature and longer residence time.

DuPont has developed the capabilities to find the best solutions to improve thin-stillage evaporation for customers, which will allow higher dry solids in the syrup while keeping the same or lower viscosity.

There is no one solution for all customers. We learned that the solution to improve thin-stillage evaporation is very dependent on feedstock and process conditions. For this reason, finding suitable improvements in Downstream Processing at a plant requires a partnership between DuPont and the customer.

Do you need to improve thin-stillage evaporation in your plant? Dupont has the capability and enzymatic tools to help you.



Nadia Ramirez Angulo, Master in Biotechnology in the University Francisco de Vitoria (Madrid). In 2012 I moved from Spain to The Netherlands to start and internship for EMEA Biorrefineries in Dupont. Based in Leiden, my work for the last two years was focus on Downstream Processing improvements for EMEA ethanol Industry, where I gain a lot of experience on viscosity reducing enzymes.

4. Biogas

4.1. **Marian Kazda**, Ulm (Germany)

Effects of sugar beet in anaerobic digestion and application in on-demand biogas production

MARIAN KAZDA¹, SHARIF AHMED¹, DANIEL EINFALT^{1,2}, KERSTIN MAURUS¹

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Readily available carbohydrates are known to spur the overall microbial activity. This effect was tested in co-digestion of sugar beet silage with fibrous substrates like grass silage to investigate the potential for increase of biogas yields. Mixtures of grass silage (G) and sugar beet silage (S) were tested in continuous laboratory-scale AD (anaerobic digestion) experiments at volatile solid ratios G:S of 1:0, 6:1, 3:1, and 1:3. It was found that even small share of sugar beet silage in 6:1 ratio increased the specific biogas and methane yields significantly in comparison to the fermentation of grass silage alone.

Besides the applicability of sugar beet silage in AD of fibrous substrates, this traditional agricultural commodity can be also used in biogas plants for on-demand electricity production. The essential advantage of biogas plants is their potential for stabilizing renewable energy supplies. The on-demand energy production is designed to meet two daily periods of high electricity consumption, i.e. during the morning and the evening hours. Thus the experimental set-up was based on hourly supply of the maize silage whilst the amount of sugar beet silage was splitted into two daily portions. Also in this approach, ratios as above of maize and sugar beet silages were tested in four 12 liter reactors run in parallel.

Both, biogas and methane production increased immediately after the input of sugar beet silage into the reactor. The high production rate lasted for about two hours and increased up to 50 % compared to periods with only maize silage supply. However, at high share of sugar beet silage, process instabilities occurred after few weeks of application. This was obvious from decreasing biogas and methane yields and acid accumulation in the reactor slurry. Besides regular measurements of process parameters also CO₂ partial pressure in the biogas slurry reacted sensitively to carbohydrate input and indicated changes in process performance.

In summary, sugar beet silage has the potential for on-demand methane production because of its high amount of fast degradable carbohydrates that can be digested easily by microorganisms and can lead also to better degradation of co-substrates.

5. Fermentation

5.1. **Ana Lopez Contreras**, Wageningen (The Netherlands)

Production of acetone, butanol and ethanol from biomass

The expected shortage of petroleum in the near future and concerns regarding the net increase of carbon dioxide emissions and environment pollution by fossil fuel combustion, have resulted in a search for sustainable sources for the production of transport fuels and chemicals. Biomass, CO₂ or syngas could provide sustainable alternatives as feedstock's for fuels and chemicals. The fermentative production of butanol, a C₄ alcohol with multiple applications as fuel or solvent, is carried out by various Clostridial species as part of the acetone butanol ethanol (ABE) process. This process is an example of a biotechnological technology with potential for production of green fuels and chemicals.

The Clostridial strains producing ABE have a wide substrate range, including C₆, C₅ and polymers such as starch. Distillers dried grains with solubles (DDGS) represent important co-product from commercial yeast fermentations, including bioethanol production from grains. In view of the current expansion of the bioethanol fermentation process, with the concomitant increase in production of DDGS, alternative applications to their main current use as animal feed are being explored.

In this study, DDGS from a bioethanol facility which uses barley as feedstock have been characterized and used as feedstock for biobutanol production. These DDGS contained, per kg of dry matter, 250 grams of protein and 390 grams of sugars, being glucose, xylose and arabinose the main sugar components. DDGS were hydrolyzed by alkaline pre-treatment

followed by enzymatic hydrolysis resulting in the solubilization of approx. 80 % of the sugars in the feedstock and contained 57 g/L total sugars. The fermentation of 20 % (w/v) DDGS suspensions and of the hydrolysate of DDGS by two acetone, butanol and ethanol (ABE)-producing bacterial strains is described. Both strains utilized the sugars in these suspensions and in the hydrolysate to produce ABE. In these cultures, the strains only utilized soluble mono- or oligosaccharides. The hydrolysate was fermentable without addition of extra nutrients, being *C. acetobutylicum* the best-performing strain, producing 8.3 g/L ABE. In addition, DDGS were used as nutrient for the fermentation of wheat straw hemicellulosic syrup (C5-syrup) with low nutrient content. This C-5 syrup was a side stream obtained from steam- exploded wheat straw, and was subjected to overliming to make it fermentable. The supplementation of the C5-syrup with DDGS eliminated the need for addition of nutrients for the fermentation.

Keywords: DDGS; Acetone-Butanol-Ethanol Fermentation; ABE Fermentation; Second Generation Feedstocks; Steam Explosion



Ana M. López Contreras is a senior scientist at Wageningen Food and Biobased Research, part of Wageningen University and Research (Wageningen, The Netherlands). She has wide experience on the production of fuels and chemicals by fermentation of second generation biomasses. She has participated or directed several projects on bioalcohol production. Current examples are EU MACROFUELS (2016-2019), EU BIOCON-CO2 (2018-2021) and the Dutch financed TKI- BBEG BioJetFuel. She has published >20 peer-reviewed scientific articles, 4 book chapters and many reports on biotechnology for fuel and chemical production, and has successfully directed 3 PhD thesis and currently supervises 2 PhD students.

5.2. **Edda Höfer**, Obrigheim/Pfalz (Germany)

Utilization of fermentation off-gas in order to extend the biorefinery concept of bioethanol plants

Detailed information will be presented at the convention.

5.3. **Cornelis Mijnders** , Utrecht (The Netherlands)

The opportunities for fungal biotech

The presentation will elaborate on the endless opportunities that are and can be created with fungi like *Aspergillus niger* and other species. Known are antibiotics and citric acid. But more and more options will become available thanks to the developments of biotechnology. Dutch DNA is active in various markets with its portfolio of fungal species. Latest developments focus on a non-viscous novel strain collection enabling the low cost production of (novel) enzymes and proteins. Enzymes are for example used for valorization of starch can be produced with the platforms DDNA is providing. DDNA has a genetic as well as process team to develop technologies from 'sequence' to a process & strain ready for upscaling. It owns 350 m2 lab facilities. DDNA is located in Utrecht/NL.



Cornelis Mijnders studied Food Science and Technology (Bsc) followed by Agricultural Economics (Msc). During his studies he developed an interest in the so called biobased economy and the efficient use of all sorts of biomass like starch, proteins and other sources to replace oil and reduce climate change and dependence of a small number of crude oil suppliers. This development creates new opportunities for agriculture, the chemical and other markets. After various positions (e.g. Ministry of Agriculture/NL, TNO) 3 years ago Dutch DNA (DDNA) was created. A spin out company from TNO further commercializing fungal biotechnology. Within Dutch DNA Cornelis is Business Development Director and focusing on the commercialization of the technologies DDNA develops, jointly with partners.

6. Analytic

- 6.1. **Sahid Babae Tooski**, Malayer (Iran)
Biomaterial based toxic gas sensor using microwave resonant cavity

Biomaterials are getting significance in the current research field of gas sensors due to great sensitivity. Performance of biomaterial based gas sensor constructed from gum Arabica and garlic extract in microwave resonant cavity had been investigated. It is shown that extract of garlic clove with multiple medicinal and chemical utility is very helpful in sensing Sulphur Oxide gas. The material under observation undergoes some momentary physical change on exposure to Sulphur Oxide gas. This change can be detected over amplified potentiometric variation through electrical circuitry of microwave resonant cavity. Manipulating this appropriate characteristic a potentiometric gas sensor of faster response and recovery time can be designed. Sensing property of the said material has been studied via microwave attenuation, reflection, and transmission.



Sahid Babae Tooski, Iran Young Researchers and Elite Club, Malayer Azad University, Malayer, Iran, (2015-present).

Project: "Size effect of nanotubes on gas sensing"

Courses: Electromagnetism Physics & Quantum Physics

• Ph.D. in Solid State Physics, Institute of Molecular Physics, Poznan, Poland, (2010-2015). Title of Ph.D. thesis: "Quantum entanglement, Kondo effect, and electronic transport in quantum dots system" Secondment for 10 months to Department of Theoretical Physics, Jozef Stefan Institute, Ljubljana, Slovenia, (September 2011 –July 2012). • Laboratory of Materials and Nanotechnology and Laboratory of Applied Physics, AmirKabir University of Technology (Tehran Polytechnic), Tehran, Iran, (2007-2010). • M.Sc. in Physics, Faculty of Physics, AmirKabir University of Technology (Tehran Polytechnic), Tehran, Iran, (2004-2007). • B.Sc. in Solid State Physics, Faculty of Sciences, Tarbiat Moallem University, Tehran, Iran, (1999-2004). Iran Young Researchers and Elite Club and Iranian Nanotechnology Intuitive Council (INIC) (2015-present) Magnetic properties and electronic transport through nanostructures, Leader: Professor Bogdan Bulka, supported by National Science Centre of Poland under the contract DEC-2012/05/B/ST3/03208 (2013-2014). As an Early Stage Researcher (ESR), Marie Curie Initial Training Network - Nanoelectronics: Concepts, Theory and Modeling (NanoCTM) since 25 October 2010 to 24 October 2013. Iranian Nanotechnology Intuitive Council (INIC) (2007-2010). Amirkabir University of Technology (AUT) (2007-2010).

7. Future

- 7.1. **Rahul Dagwar**, Pune (India)
Smart solutions for sustainable ethanol production: How to increase performance of your existing ethanol plants?



Rahul Dagwar, graduated in chemical engineering with diploma in business administration. Working with Praj Industries for more than 10 years. Praj is a global leader in providing solutions for biofuels industry. In Praj I am responsible for business development in Europe. Experience in process design and commissioning of grain based ethanol plants which includes large scale ethanol plants in Europe

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2. Raw material

- 11³⁰ 2.1. **Arjen van Tuijl**, Leiden (The Netherlands)
Processing beyond the limits of wheat ethanol manufacturing

3. Enzymes

- 12⁰⁰ 3.1. **Nadia Ramirez Angulo**, Leiden (The Netherlands)
Enzymatic tools for improvements in thin-stillage evaporation

12³⁰ Lunch Break

4. Biogas

- 13³⁰ 4.1. **Marian Kazda**, Ulm (Germany)
Effects of sugar beet in anaerobic digestion and application in on-demand biogas production

5. Fermentation

- 14⁰⁰ 5.1. **Ana Lopez Contreras**, Wageningen (The Netherlands)
Production of acetone, butanol and ethanol from biomass
- 14³⁰ 5.2. **Edda Höfer**, Obrigheim/Pfalz (Germany)
Utilization of fermentation off-gas in order to extend the biorefinery concept of bioethanol plants

15⁰⁰ Communication Break

- 15³⁰ 5.3. **Cornelis Mijnders**, Utrecht (The Netherlands)
The opportunities for fungal biotech

6. Analytic

- 16⁰⁰ 6.1. **Sahid Babae Tooski**, Malayer (Iran)
Biomaterial based toxic gas sensor using microwave resonant cavity

7. Future

- 16³⁰ 7.1. **Rahul Dagwar**, Pune (India)
Smart solutions for sustainable ethanol production: How to increase performance of your existing ethanol plants?

- 17⁰⁰ **Closing remarks**
by the Chairman of the Starch Experts Group of the Association of Cereal Research, **Willi Witt**, Oelde (Germany)

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