



EUROFOODWATER

BOOK OF ABSTRACTS

7th International Conference on

Water in Food

*Scandic Continental, Helsinki, Finland
June 3–5, 2012*

Organised by



HELSINGIN YLIOPISTO
HELSINGFORS UNIVERSITET
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Good Food, Good Life



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Sunday, 3rd of June

17:00–19:00	Registration
19:00–19:05	Welcoming words
19:05–19.45	Plenary Lecture PL1
19:45–21:00	Get-together party

Monday, 4th of June

8:30–8:40	Opening of the conference
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Session I: Water and Physical State of Food

8:40–9:20	Plenary Lecture PL2
9:20–9:40	Oral Communication OC1
9:40–10:00	Oral Communication OC2
10:00–11:00	<i>Coffee break and posters & exhibition</i>
11:00–11:20	Oral Communication OC3
11:20–11:40	Oral Communication OC4
11:40–12:00	Oral Communication OC5
12:00–12:20	Oral Communication OC6
12:20–13:40	<i>Lunch</i>

Session II: Water and Stability of Food

13:40–14:20	Plenary Lecture PL3
14:20–14:40	Oral Communication OC7
14:40–15:00	Oral Communication OC8
15:00–16:00	<i>Coffee break and posters & exhibition</i>
16:00–16:20	Oral Communication OC9
16:20–16:40	Oral Communication OC10
16:40–17:00	Oral Communication OC11
17:00–17:20	Oral Communication OC12
20:00–	<i>Conference Dinner</i>

Tuesday, 5th of June

Session II: Water and Stability of Food (*Continue*)

- 9:00–9:20 Oral Communication **OC13**
9:20–9:40 Oral Communication **OC14**
9:40–10:00 Oral Communication **OC15**
10:00–10:20 Oral Communication **OC16**
- 10:20–11:20 *Coffee break and posters & exhibition*

Session III: Water and Quality of Food

- 11:20–11:40 Oral Communication **OC17**
11:40–12:00 Oral Communication **OC18**
12:00–12:20 Oral Communication **OC19**
- 12:20–13:40 *Lunch*
- 13:40–14:00 Oral Communication **OC20**
14:00–14:20 Oral Communication **OC21**
14:20–14:40 Oral Communication **OC22**
14:40–15:00 Oral Communication **OC23**
- 15:00–16:00 *Coffee break and posters & exhibition*
- 16:00–16:40 *Oral presentation of selected posters*
- 16:40–17:00 *Concluding remarks and closing of the conference*

Scientific Programme
7th International Conference on Water in Food
June 3–5, 2012

Sunday, 3rd of June

- 19:00–19:05 Dr. Kirsi Jouppila (University of Helsinki)
Welcoming words
- 19:05–19.45 **PL1:** Professor Theodore P. Labuza (University of Minnesota)
“Water activity, a short history of the key players in the evolution of free vs bound water to how we view water today as a physical chemistry concept”

Monday, 4th of June

- 8:30–8:40 Vice-Rector, Professor Jukka Kola (University of Helsinki)
Opening of the conference

Session I: Water and Physical State of Food

Chaired by Prof. Mohamed Mathlouthi (University of Reims)

- 8:40–9:20 **PL2:** Professor Yrjö H. Roos (University College Cork)
“Critical aspects of water plasticization of food solids”
- 9:20–9:40 **OC1:** Fundo F.J., Quintas M.A.C. and Silva C.L.M.
“Molecular mobility and the thermomechanical properties of chitosan films”
- 9:40–10:00 **OC2:** Kurek M., Galić K., Voilley A. and Debeaufort F.
“Influence of relative humidity on the functional and barrier properties of active biopolymer packaging films”
- 10:00–11:00 *Coffee break and posters & exhibition*
- 11:00–11:20 **OC3:** Renzetti S. and Voogt J.A.
“Fundamental insights in moisture migration with industrial relevance: a quantitative high-throughput screening system for water migration in multi-texture foods combined with a predictive model of shelf-stability”

- 11:20–11:40 **OC4:** Lee Y.-C., Huang S.-R. and Chen H.-W.
“Effects of water mobility on heating process of porous starchy foods”
- 11:40–12:00 **OC5:** Schutyser M., Perdana J., Fox M. and Boom R.
“Single droplet drying of heat sensitive food ingredients – Establishing inactivation kinetics”
- 12:00–12:20 **OC6:** Nowacka M., Tylewicz U., Laghi L., Dalla Rosa M. and Witrowa-Rajchert D.
“Effect of ultrasound treatment on the water state in kiwifruit during osmotic dehydration”
- 12:20–13:40 *Lunch break*

Session II: Water and Stability of Food

Chaired by Prof. *Heinz Isengard* (University of Hohenheim)

- 13:40–14:20 **PL3:** Professor Stephan Drusch (Technische Universität Berlin)
“The role of water in encapsulation of food ingredients”
- 14:20–14:40 **OC7:** Zhou Y. and Roos Y.H.
“ α -tocopherol stability in water plasticization of spray-dried and freeze-dried emulsions”
- 14:40–15:00 **OC8:** Labuza T.P. and Ismail B.
“Changes in stability of bioactive peptides through glycation with short dextrans”
- 15:00–16:00 *Coffee break and posters & exhibition*
- 16:00–16:20 **OC9:** Jouppila K.
“Effect of thermal and water plasticization on crystallization behavior of various sugars”
- 16:20–16:40 **OC10:** Yazdanpanah N. and Langrish T.A.G.
“Role of the crystalline outer-layer in maintaining the physico-chemical properties of milk powder during storage”
- 16:40–17:00 **OC11:** Potes N. and Roos Y.H.
“Glass transition and lactose crystallization by water plasticization in static and dynamic studies of lactose-maltodextrin systems”
- 17:00–17:20 **OC12:** Mauer L., Ghorab M. and Taylor L.
“Effects of co-formulation on the water-solid interactions of deliquescent crystalline and amorphous ingredients”

Tuesday, 5th of June

Session II: Water and Stability of Food (Continue)

Chaired by Prof. *Stephan Drusch* (Technische Univ. Berlin)

- 9:00–9:20 **OC13:** *Sacchetti G., Neri L., Laghi L., Capozzi F. and Pittia P.*
“Multidisciplinary approach for the evaluation of the effect of water status and mobility on the activity of peroxidases in solution”
- 9:20–9:40 **OC14:** *della Campa M., Lo Scalzo R., Bertolo G. and Cattaneo T.*
“A multi-purpose industrial sugar substitute from grape obtained in a short supply chain context”
- 9:40–10:00 **OC15:** *Mathlouthi M. and Rogé B.*
“Water–sucrose interactions and the mechanism of sugar bloom formation in frozen icing”
- 10:00–10:20 **OC16:** *Puolanne E. and Halonen M.*
“Theoretical aspects of water-holding in meat”
- 10:20–11:20 *Coffee break and posters & exhibition*

Session III: Water and Quality of Food

Chaired by Prof. *Yrjö H. Roos* (University College Cork)

- 11:20–11:40 **OC17:** *Bell S. and Carroll P.*
“Metrologically robust moisture measurements”
- 11:40–12:00 **OC18:** *Federle H.*
“New developments of online NIR measurement in food processes”
- 12:00–12:20 **OC19:** *Hădărugă D.I., Hădărugă N.G., Corpaş L. and Isengard H.-D.*
“Karl Fischer water titration of cyclodextrins: parameter optimization”
- 12:20–13:40 *Lunch break*

- 13:40–14:00 **OC20:** Heinonen M.
“Traceability of water activity measurements”
- 14:00–14:20 **OC21:** Meunier V. and Vuataz G.
“ a_w of amorphous food solids: equilibrated versus apparent values”
- 14:20–14:40 **OC22:** Carter B.P. and Buehler M.G.
“Phase changes observed using dielectric spectroscopy while scanning either temperature or water activity”
- 14:40–15:00 **OC23:** Bernasconi M.
“How water activity can reduce food waste and improve product quality”
- 15:00–16:00 *Coffee break and posters & exhibition*
- 16:00–16:40 Oral presentation of selected posters
Chaired by Dr. *Kirsi Jouppila* (University of Helsinki)
- 16:40–17:00 Concluding remarks and closing of the conference
Professor Heinz Isengard (University of Hohenheim)

EFW 2012
ORAL PRESENTATIONS



Water activity, a short history of the key players in the evolution of free vs bound water to how we view water today as a physical chemistry concept

Professor Theodore P. Labuza

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In 1953 an Australian (Walter Scott) on leave from CSIRO and in England, met John Pitt who talked about a concept of ERH, i.e. Equilibrium Relative Humidity. Scott took that idea and showed that it was the ERH that controlled mold growth on different foods in a unique set of experiments. This idea expanded into research on water mobility using NMR (Denise Simatos), using radio labeled molecules (Duckworth) and on chemical reactions (Karel). From there in the next 15 years a number of researchers who worked on water in foods met at a meeting in 1965 in Scotland where they put forth the idea of a re-occurring meeting on water called the International Symposium on the Properties of Water (ISOPOW). It is through those meetings (the 11th was held in Mexico in 2010) that a body of principles has taken food science out of the “cook and look” methods of analysis and food formulation to where we now have an established set of scientific paradigms to help solve processing and packaging problems such that we can ensure suitable quality and shelf life. This presentation will briefly review those parameters in an historical sense which have moved food science and food engineering into the 21st century.

These paradigms include:

1. Microbial growth limitations as a function of ERH or water activity (a_w)
2. The measurement of moisture sorption isotherms
3. New means to measure water activity and moisture content
4. The influence of composition and temperature on isotherms
5. Use of isotherms for prediction of moisture gain/loss of packaged foods
6. Moisture loss/gain and textural changes in foods
7. Water activity migration principles in multidomain foods
8. The use of NMR to study moisture migration.
9. The concept of water activity as a thermodynamic driving force vs. the free vs. bound water concept.
10. Degradation rates of chemical/enzymatic reactions as a function of a_w
11. The stability map of food degradation
12. The hurdle concept and intermediate moisture foods – a new category of processed food.
13. Powder state changes and water: stickiness, caking and collapse
14. The concept of glass transition and influence of ingredients
15. The State Diagram: combining a phase diagram with the T_g curve

Critical aspects of water plasticization of food solids

Professor Yrjö H. Roos

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Cork, Ireland

Water in foods has a significant importance to physical state of solids, reaction rates, microbial growth and shelf life. Water activity, a_w , water content and water plasticization measurements often use simplistic approaches and assumptions of validity of data as global measures of food stability. Similar approaches have been taken in glass transition measurements. Such measurements of a_w give excellent reference values for microbial safety as the data apply across all components at equilibrium. Water content, water plasticization and glass transition measurements also give global values for a food, but significant differences within microstructure in water distribution and state of components are expected, i.e., the local water contents and individual water plasticization of food components become more important determinants of physicochemical properties and reaction rates than the global parameters.

Water plasticization has been studied extensively using single food components, primarily carbohydrates. These studies have shown a substantial effect of water on the glass transition temperature, T_g . Water content dependence of the T_g is important to food processing and stability at various temperatures. The mole fraction of water reduces water activity and for small molecular weight glass formers an almost linear relationship for T_g against a_w can be found. Critical values for a_w and water content may be used to refer to glass transition occurring at a standard temperature, but measurements of a_w , water content or T_g do not describe rates of changes if the critical values are exceeded. A typical approach to evaluate effects of the parameters on rates of changes above their critical values has been an application of the Williams-Landel-Ferry (WLF) relationship to describe temperature dependence of relaxation times above the T_g . Classification of solids to fragile and strong glass formers has also been proposed. Angell plots of relaxation times against T_g/T in such classification show serious limitations for comparison of glass formers with different T_g values, because only the T_g/T point (glass transition) can be defined and for $T_g/T < 1$ fragilities must differ. Water plasticization of food components decreases the T_g and fragilities at various water contents for the same glass former become problematic. We present a WLF-based “fluidness” characterization of food components that allows comparison of relaxation times above the T_g at various water contents.

Data on lactose crystallization suggest that water plasticization of the carbohydrate phase is required to mobilize sugar molecules, but the rate of crystallization and structural relaxation times may become decoupled in complex systems. Rates of reactions of entrapped and encapsulated components may have a complex dependence on reactant state and diffusion, but show increasing rates with decreasing relaxation times resulting from water plasticization to above critical values of the encapsulant matrix. Critical data of water plasticization of food solids to food formulation, solids characterization and shelf life evaluation are often those of the most water sensitive components, but structural relaxation times may show complex relationships with solids properties and stability.

Molecular mobility and the thermomechanical properties of chitosan films

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Edible and biodegradable films have been successfully explored at experimental level, attracting interest in the food preservation and packaging technology fields. Chitosan is a biopolymer and is considered as being biocompatible, biodegradable and possesses antimicrobial activity and filmogenic properties and for that has been thoroughly used in such studies.

In this work, we aim to describe the link between the water and the plasticizant mobility in the partially crystalline polymeric structure of chitosan films and the measured thermo physical properties.

For that, film forming solutions of 1, 2 and 3% w/v of chitosan with different levels of plasticizing agent (glycerol 10, 50 and 90% w/v) were prepared and films were produced by solvent casting. Films thermal properties (T_g and Δh) were determined using differential scanning calorimetry (DSC). Mechanical properties (elongation at break and tensile strength) were evaluated in extension with an Instron Universal Testing Machine. Mobility was assessed by the water and glycerol relaxation times, studied using a resonance magnetic technique (NMR).

In general, results show that, as is described by theory, samples glass transition (T_g) decrease while molecular mobility increases. As for crystallinity, evaluated by Δh , increased with increasing water and glycerol mobility, showing that once the polymeric chains are organized in the crystalline form, the interaction polymer/ plasticizing is minimized. Mechanical properties evaluated (EB and TS) also followed the tendency described in literature: increase of mobility means an increase of EB and a decrease of TS. A anti-plasticizing effect was previously described when solutions where prepared with 2% w/v of chitosan and 50% w/v glycerol, such effect was also evident in the mobility of water and glycerol in those films.

These set of results demonstrate how mobility could be a crucial aspect to evaluate food systems physical and quality parameters.

Influence of relative humidity on the functional and barrier properties of active biopolymer packaging films

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Foods susceptible to microbial and moisture damage need to be packed in materials with special functional and barrier properties. The effectiveness of antimicrobial films is dependent on the release rate of the active compounds, either directly to food surface or via air. The aim of this study was to determine the influence of relative humidity (RH) (0, 75 and >98%) at 3 different temperatures (4, 25 and 37 °C) on the carvacrol release from chitosan film used for preservation of food with high water activity. Moreover, gas and water vapour barrier film properties were measured as a function of relative humidity. The release of carvacrol was significantly increased at >98% RH during the 60 days of storage. The most important release was measured at 37 °C (99.8%) followed by 98.7% and 97.8% at 20 and 4 °C, respectively. The lowest release was measured at 4 °C and dry atmosphere (16.9%). Hence the active compound release was more influenced by water vapour than the temperature. The study of the water vapour permeability (WVP) was realized at 25 °C and for three relative humidity differentials (30–100%, 30–75%, 0–33%). Carvacrol significantly decreased the WVP for the higher gradients, but had opposite effect at weaker. The presence of water vapour significantly increased the sensitivity of both hygroscopic chitosan films with/or without carvacrol. Still in the presence of water, carvacrol decreased the WVP compared to pure chitosan film. This suggests a plasticizing effect of carvacrol, only noticeable for the lowest RH gradient, because at the lower water content the plasticizing effect of water was less marked.

This study showed importance of water on the functional properties of chitosan active film that can increase the product shelf-life and improve its quality.

**Fundamental insights in moisture migration
with industrial relevance:
A quantitative high-throughput screening system
for water migration in multi-texture foods combined
with a predictive model of shelf-stability**

Renzetti S. and Voogt J.A.

TNO, Expertise Group Functional Ingredients, Zeist, The Netherlands

The development of shelf-stable multi-texture food products is technologically challenging due to water migration occurring between the moist (high a_w) and dry (low a_w) components, which results in a rapid loss of the textural and sensorial properties of the product. Insights into the influence of morphology and composition of the multi-texture food components on the kinetics of the water migration are therefore important to guide technologists in the development of shelf-stable products. Edible barriers are a suitable technological solution in order to limit water migration between food components and hence improve shelf-stability. However, the development of functional barrier solution with industrial relevance is often challenging, especially in high moisture foods, due to differences in barrier performance between commercial applications and conventional laboratory tests. We present here a high-throughput screening system developed to gain quantitative information with regards to the kinetics of moisture diffusion in a bi-component food model representative of a real product. The kinetic parameters obtained on water diffusion through the food components and an edible barrier are combined together with key material properties of the components in a predictive model. The model provides information on the moisture/ a_w profiles of the components during storage. The methodological approach presented combines fundamental insights with applied relevance, thus enabling fast and efficient development of stable multi-texture foods.

Effects of water mobility on heating process of porous starchy foods

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Porous media are commonly used as insulation materials for their low thermal conductivities. On the contrary, steaming of porous foods shows a very high temperature increasing rate that might be due to the enhancement by hot water and vapor diffusion.

Our objective was to investigate the influence of water mobility, in forms of liquid and vapor, on increasing heat transfer rate in heating of a porous starchy food, Chinese steamed bun. To achieve this objective, steam buns were heated in a steamer and a convective oven, temperature profiles at various locations of the bun and weight changes during the process were measured. Some samples were wrapped with polyethylene (PE) film as a barrier to identify effects of surface water and vapor transport. Mathematical models were developed that consider coupled energy, water and vapor transport and were solved using finite element software (COMSOL Multiphysics). The models predicted temperature profiles and weight changes were validated with experimental observations.

Mathematical simulation results explain that during steaming diffusion of absorbed condensate will increase the temperature elevation rate in the outer region and evaporation–condensation mechanism of vapor contributes to a steep sigmoid-shaped temperature increasing profile in the core region of the bun. The characteristic of steaming and oven heating processes can also be well distinguished by the mathematical models.

Based on finite element method, the coupled multi-physics modeling validated the experimental findings. We can conclude, therefore, that effects of water mobility is the key to enhance the heat transfer of porous starch foods.

Single droplet drying of heat sensitive food ingredients – Establishing inactivation kinetics

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Heat sensitive food ingredients may suffer from severe activity loss during spray drying. The inactivation is dependent both on temperature and water content during drying. In our research both constant heating experiments and single droplet drying experiments are performed to unravel inactivation of β -galactosidase (acting as the model ingredient) as a function of temperature and water content. The single droplet drying method consists of a pneumatic micro-dispenser for droplet generation and a drying chamber for controlled drying. During drying, the droplet is located on a hydrophobic surface to retain its spherical shape. In essence, this single droplet drying method mimics the drying of a droplet during spray drying (Perdana *et al.*, 2012). A mass and heat transfer model is developed to accurately describe the drying history of the deposited droplet (i.e. specifically the spatial distribution of water content in the drying droplet), while a kinetic model is used to describe the inactivation of β -galactosidase as a function of temperature and water content. A novel kinetic model was developed first on the basis of constant heating experiments. Subsequently, the kinetic model was used to obtain kinetic inactivation parameters from single droplet drying data directly. This approach is practical for the development of a high throughput experimentation method on spray drying. The idea is that the single droplet screening platform can be used to systematically screen for dehydration strategies to reduce heat damage, e.g. by using specific drying conditions, by introducing a protective matrix formulation (glass forming carbohydrates), or by incorporating specific pre-treatments (e.g. probiotic strain selection).

Perdana *et al.*, *Food Bioprocess Technol* (2012) DOI 10.1007/s11947-011-0767-4

Effect of ultrasound treatment on the water state in kiwifruit during osmotic dehydration

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Osmotic dehydration (OD) is a widely used method for partial water removing by immersion of cellular tissue in hypertonic solution. Ultrasound waves applied before OD treatment could improve this process, however little is known about the effect of ultrasound on the water state in osmotic dehydrated fruits. For this purpose, this work presents the water mobility study in kiwifruit slices treated with ultrasound before osmotic dehydration.

Kiwifruits (*Actinidia deliciosa var deliciosa* cv Hayward) were peeled and cut into 10 mm thick slices and then subjected to ultrasonic waves in the bath at a frequency of 35 kHz for 10, 20 and 30 minutes. The osmotic dehydration was carried out by dipping the samples in 61.5% sucrose solution equilibrated at 25 °C for pre-established contact period of 0, 10, 20, 30, 60 and 120 min. The water mobility, in terms of transverse relaxation time (T_2) and peak intensity, were analyzed at Low Frequency Nuclear Magnetic Resonance (LF-NMR). The LF-NMR measurements were performed at 24 °C with the CPMG pulse sequence using a Bruker Minispec PC/20 spectrometer operating at 20 MHz.

Ultrasound treatment changed the mobility of water in different compartments (vacuoles, cytoplasm/extracellular spaces, cell wall) of kiwifruit slices. The most relevant changes were observed in samples treated only with 30 min ultrasound. Tissue submitted to ultrasound treatment and 120 min of OD was characterized by lower water proton pool mobility (T_2) in vacuoles and cytoplasm/extracellular spaces in comparison to fresh tissue. Higher intensity of water proton pools was observed in cytoplasm and extracellular spaces, while in vacuoles this parameter decreased. This phenomenon indicates the water from vacuoles goes into cytoplasm and extracellular spaces.

The role of water in encapsulation of food ingredients

Professor Stephan Drusch
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A variety of food ingredients like aroma-active compounds, nutritional oils, natural food colorants, vitamins or antimicrobial extracts are encapsulated prior to their use in food production. All these food ingredients have in common, that they are isolated from their natural matrix and are susceptible against environmental factors like light or oxygen. As a consequence changes in the chemical structure of key components may occur, which result in a loss of functionality (flavour, colour, nutritional value). Thus, encapsulation is a common strategy to protect food ingredients and to control their release.

Water, on one hand, is the solvent or the continuous phase in dispersed encapsulation systems and enables the formation of structural elements in the encapsulating matrix. Incompatibility of polymers in aqueous solution is the driving force in complex coacervation for encapsulation. In the case of a solvent-activated release mechanism, contact with water is the trigger for release of the encapsulate.

On the other hand, dissolution upon contact with water frequently limits the range of applications for the encapsulated food ingredient. Resistance against dissolution and swelling might be desirable to obtain sensorical effects after ingestion of foods or to control delivery of functional ingredients *in vivo*. Like for a variety of foods, water needs to be removed from encapsulation systems to increase the stability against microbial spoilage. Materials science aspects as well as process engineering parameters need to be taken into consideration to maintain the desired physical structure and functionality of the encapsulation system. Finally, water is responsible for adverse changes like phase transition of the capsule matrix components and structural changes, which affect molecular mobility and as a consequence limit the stability of the encapsulate. Coating might be used to prevent moisture sorption and to further modify the encapsulating matrix.

α -tocopherol stability in water plasticization of spray-dried and freeze-dried emulsions

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Dehydration produces food materials with encapsulated sensitive components at low water contents. Stability of sensitive components is often dependent on the dehydration method and encapsulants. The objectives of the present study were to investigate stability of α -tocopherol in dehydrated systems and physical properties of the encapsulants. Spray-dried and freeze-dried lactose-soy protein-oil (3:1:2), trehalose-soy protein-oil (3:1:2), lactose-trehalose-soy protein-oil (3:3:2:4) systems contained 1.7% of α -tocopherol (w/w of solids). The stability of α -tocopherol at 0.33 water activity under vacuum in dark was monitored during storage at 20–55 °C up to 60 d. The solids were characterized for water sorption and state transitions. The emulsion properties were followed in reconstitution during storage. Spray-drying and freeze-drying retained 80% and 100% of α -tocopherol, respectively. α -tocopherol was stable in dehydrated systems below (20 °C) and around (30 and 37 °C) the glass transition of the solids during 60 d of storage and the zeta-average of reconstituted materials showed very small increases in all systems. Above the glass transition, freeze-dried systems showed full retention of α -tocopherol up to 55 d, except for the lactose-soy protein-oil system. Loss of α -tocopherol in spray-dried systems occurred at 55 °C at 12 d to 30 d and an increase of zeta-average was observed. The loss of α -tocopherol was in agreement with structural changes, including crystallization, in the dehydrated emulsions. The results indicated the importance of the processing methods and parameters on the physical properties of the encapsulants and the stability of sensitive components.

Changes in stability of bioactive peptides through glycation with short dextrans

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Moisture-induced protein/peptide aggregation is a significant quality challenge for the \$3-billion baby formula (dry food) market and the \$1-billion nutrition/energy bar (intermediate-moisture food, IMF) market in the US. Such aggregation causes loss of both acceptability and nutritional quality. Characterizing this moisture-induced effect will aid in the development of technologies that will limit deterioration of quality and the consequent challenges hindering the growth of functional protein hydrolysates (bioactive peptides). Our objective is to investigate the effect of Maillard-induced glycosylation on the preservation of structural and nutritional quality of bioactive hydrolysates. We recently showed that both a_w and T_g are good indicators of the onset of physical changes leading to peptide aggregation in an egg white hydrolysate system. We also found that moisture-induced protein/peptide aggregation could occur in IMF matrices containing whey protein hydrolysates during storage. Variations in protein/peptide sources caused variations in the kinetic rate of changes in microstructure and texture during storage. To prevent aggregation, Maillard-induced glycosylation was employed using whey protein as a model. Several physicochemical/structural changes, including resistance to denaturation, a shift to more acidic pI, reduced exposure of sulfhydryl and hydrophobic groups, shift of α -helix secondary structure to β -sheet, and unique glycosylation sites, contributed to reduction in protein aggregation. Other food protein isolates and their hydrolysates will be studied. Several advanced analytical techniques are employed (e.g. Raman) to characterize the structural changes during storage as a function of moisture content using dry food and IMF matrices. We will subject the hydrolysates to Maillard-induced glycosylation and evaluate structural changes and digestibility using the same proposed matrices. This research will lead to new strategies for preserving quality and enhancing storage stability of protein-based dry foods and IMF.

Keywords: moisture-induced protein/peptide aggregation, protein hydrolysates, Maillard-induced glycosylation

Effect of thermal and water plasticization on crystallization behavior of various sugars

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Crystallization may occur in amorphous sugar due to thermal and water plasticization if the glass transition temperature (T_g) of amorphous sugar decreases to below storage temperature. Under such temperature and relative humidity (RH) conditions crystallization is enabled due to increased molecular mobility in material. The aim of the present study was to compare crystallization behavior of various disaccharides as detected from the loss of adsorbed water using either a static or dynamic analysis. Amorphous disaccharides were produced by freeze- or spray-drying and were stored under temperature and relative humidity (RH) conditions resulting in crystallization. Loss of water was detected gravimetrically by weighing the samples stored in vacuum desiccators over saturated salt solutions at intervals during storage (static analysis) and using Dynamic Vapor Sorption (DVS) Intrinsic instrument (dynamic analysis). Physical state of amorphous disaccharides was analyzed using differential scanning calorimeter (DSC).

The rate of crystallization of various disaccharides was observed to increase with increasing RH and temperature difference between storage temperature and T_g . Start and progress of crystallization were easy to follow in samples showing clear steady-state water contents prior to the gradual loss of adsorbed water. However, steady-state water contents were not achieved under higher RH and temperature conditions due to simultaneous water adsorption and water desorption. Under lower RH and temperature conditions crystallization did not occur within a reasonable time. Thus, selecting proper conditions for crystallization is important.

Knowledge of effect of the thermal and water plasticization on changes occurring in amorphous sugars is important in selection of proper processing and storage conditions in order to maintain desired properties of food products containing amorphous sugars such as food powders and encapsulated starter cultures and probiotics.

Role of the crystalline outer-layer in maintaining the physico-chemical properties of milk powder during storage

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Hygroscopicity and high permeability in amorphous materials accelerate the deteriorative changes, caused by high water activity, in food powders during long storage. Amorphous materials, in this research Skim Milk Powder, have higher moisture sorption rate and greater permeability, less stability, better rehydration/dissolution behavior and more bioavailability compare to crystalline materials. Flowability (less stickiness) and stability of milk powder may be improved by crystallization of the amorphous contents of the powders, but it has influences on the functional properties of the powder (such as dissolution rate) and decreases the molecular stability of the proteins in the matrix.

The recently developed milk powder with egg-shell structure particles, which are individually wrapped in a thin layer of crystalline lactose, preserves good stability and functionality in the fresh semi-crystalline state. This edible film, formed from the inherent ingredients, has less permeability and moisture sorption and can protect the internal active ingredients of the milk powder against deteriorative changes influenced by increasing the water activity during storage.

The rationale of this research has been to compare the high water activity derived changes in the milk powders with the conventional and the egg-shell structure during a six month storage time. The “micro-packaged” particles showed lower changes in the surface composition, protein modification and crystallinity during long term storage at 25–30°C and 35% RH.

Glass transition and lactose crystallization by water plasticization in static and dynamic studies of lactose-maltodextrin systems

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Crystallization of sugars is delayed in amorphous mixtures with maltodextrins (MD). Maltodextrins hinder diffusion and reduce molecular mobility by increasing the glass transition temperature, T_g . The objective of the present study was to investigate static and dynamic water sorption and lactose crystallization properties in lactose-MD systems.

Freeze-dried solids of lactose and lactose-MD were prepared from solutions containing 20% (w/w) solids. MDs with low (9–12) and high (23–27) DE were mixed with lactose at 10:90, 20:80, and 30:70, and 60:40 (w/w) ratios. Time-dependent sorbed water contents in the amorphous materials were followed. The lactose-MD at 90:10, 80:20, 70:30, and 40:60 ratios were stored over saturated salt solutions (11.4–76.1% relative humidity, RH) in vacuum desiccators at 20 ± 2 °C for 25 days in gravimetric studies. Dynamic water sorption (AquaSorb, Decacon, Pullman, WA, U.S.A.) was used to determine effects of MD in lactose-MD systems. The Guggenheim-Anderson-de Boer (GAB) equation was fitted to amorphous solids data to model water sorption of lactose-MD systems in the absence of crystalline components. The T_g of the lactose-MD systems were measured using DSC (0–44.1% RH).

Lactose crystallization was absent at low RH and experimental water sorption (11.4–44.1% RH) data were obtained. We found that steady state sorbed water contents in the absence of crystallization at all humidities were the sum of those of amorphous components. Water sorption data of each component could be derived from total water sorption of mixtures over a wide RH range, including higher humidities (54.5–76.1% RH), i.e., amorphous sorption isotherm for crystallizing lactose could be obtained from non-crystalline lactose-MD systems. Dynamic water sorption behavior of mixed systems was different from lactose and water plasticization over the experimental RH range did not result in glass transition in all systems. Crystallization of lactose and lactose in lactose-MD systems at 25 °C occurred only at high a_w , above critical water activity and content values, in the dynamic studies. Crystallization of lactose during dynamic water sorption was absent in lactose-MD systems at 70:30 and 40:60 ratios. The lactose-high DE MD (80:20 and 70:30 ratios) systems showed a higher stability against crystallization of lactose up to 0.85 a_w . This result was in agreement with gravimetric studies. The results also showed that MD with high DE inhibited lactose crystallization more effectively than low DE MD. The water content of lactose-MD at 40:60 ratio (54.5–76.1% RH) from the gravimetric study provided water sorption data of amorphous lactose and lactose-MD systems, and predicted full water sorption isotherms of amorphous lactose and lactose-MD systems over a wide range of RH.

Effects of co-formulation on the water-solid interactions of deliquescent crystalline and amorphous ingredients

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The influence of deliquescent crystalline materials on the hygroscopicity and glass transition (T_g) of amorphous maltodextrins (MDs) as well as the impact of amorphous solids on the deliquescence of the crystalline solid in binary mixtures were determined. Binary amorphous:crystalline mixtures (1:1 w/w) were prepared from four MDs with varying molecular weights (MW) and three crystalline materials - sucrose (S), fructose (F), and sodium chloride (NaCl). Formulations and storage conditions were chosen such that data were collected for samples stored below both T_g and deliquescence (RH_0), above T_g but below RH_0 , above RH_0 but below T_g , and above both T_g and RH_0 . The deliquescence points (RH_0) of the crystalline materials and their binary mixtures with MDs were determined using three techniques: Decagon Aquasorb, Decagon VSA, and Projekt Messtechnik SPS. All ingredients and blends were stored in desiccators of different %RH (44%, 63%, 75%, 85% and 98% RH) at 22°C until the equilibrium moisture content (EMC) was attained. The glass transition temperature (T_g) of the amorphous materials was determined by differential scanning calorimeter (DSC) from the 2nd scan of these equilibrated samples. Dissolution of single crystalline particles and particles in contact with a MD at a controlled RH lower than the RH_0 was monitored by polarized light microscopy (PLM) and second order nonlinear optical imaging of chiral crystals (SONICC). The presence of crystalline ingredients lowered the T_g of MDs, although the trends varied by formulation. All data revealed that the dissolution of crystalline ingredients occurred at lower RHs than their individual RH_0 s when MDs were present, and lower MW MDs initiated dissolution at lower RHs than higher MW MDs. Thus, co-formulating deliquescent crystalline and amorphous solids could induce both the glassy-rubbery phase transition and dissolution/ deliquescence of the crystalline ingredients at lower RHs than expected. This alters the moisture sorption behaviors of the blends, resulting in synergistic and not additive moisture sorption patterns.

Multidisciplinary approach for the evaluation of the effect of water status and mobility on the activity of peroxidases in solution

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The aim of this study was to investigate the effect of water status as described by water activity (a_w) and freezable water and other physical properties of the system, such as bulk viscosity, transverse relaxation time and glass transition temperature (T_g'), on the activity of horseradish peroxidase (HRP) and bovine lactoperoxidase (LPO) in buffered solutions. To this purpose, co-solutes such as sugars and maltodextrin were used to modulate the a_w , viscosity and T_g' of the solutions.

HRP (44,173.9 Da) is an enzyme with an highly accessible active site whilst LPO is a larger enzyme (78,431 Da) with a deeply buried active site with a restrictive substrate access channel which should limit the influence of environmental factors on enzymatic catalysis.

In viscous solutions characterized by different composition the inhibition of the HRP activity was dependent both on the water status and on the mobility of the system, as described by the inverse of viscosity and $T-T_g'$. Viscosity was the most important factor in the inhibition of enzymatic activity in solutions characterized by the same T_g' value, but when T_g' was changed, due to changes in the solutes composition, the latter became a key factor in the regulation of the enzyme activity. HRP activity was much more influenced by changes in the water status than LPO activity which, in turn, was much more influenced by changes in viscosity. The results of this study suggest that the translational diffusivity of the enzyme which, according to the Stokes-Einstein law, is inversely related to its molecular weight, is the most critical factor in enzymatic catalysis in solutions.

A multi-purpose industrial sugar substitute from grapefruit obtained in a short supply chain context

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Modern food industry is constantly looking for new paths in order to obtain original products. More and more often the attention during their development is focused on their healthiness and naturalness. In this context small producers are penalized due to safety problems. Recently, research on small food processing plants is giving new tools to small and craft producers. It is now possible to combine food safety and quality, typical of big factory, with high quality raw materials, typical in craft producer.

Within the “MIERI” project, a national research project baked by the Italian Ministry of Agriculture (acronym meaning: realization of small scale equipment for food processing, also working with renewable energy), new products were “tailored” on a miniaturized food processing line, projected and built in a previous part of the above-mentioned project.

The aim of this work is the study of an industrial product able to sweeten, colour, acidify and preserve the original bioactive natural compounds.

The product (a honey-similar high viscosity fluid) is obtained by concentrating under vacuum a clarified white and black grape must obtained from blanched or not-blanched berries. A potential use of this grape-derived is in yogurt and ice-cream industry.

The analytical approach was a comparison between blanched (T) and untreated samples (NT). The antioxidant power (quantified by Folin-Ciocalteu index) and the total polyphenols content were made by spectrophotometry; the sugar content was made by HPLC and refractometry, and water activity was made by a dew point water activity meter. The most important finding was that the blanching pre-treatment of grape berries, in comparison with not-blanched ones, strongly increased the phytochemicals content of both white and black grape-derived product.

Water–Sucrose interactions and the mechanism of Sugar bloom formation in frozen icing

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Sugar bloom is observed on the surface of pastry such as éclair coated with icing sugar preparation during freezing. The sucrose hydrates which correspond to the bloom at the surface of frozen pastry rapidly dissolve as temperature increases and give rise to unpleasant aspect of craters at the surface of the frozen material.

The formula of fondant constituting the basics of icing matrix generally involves about 88–89% of carbohydrates, including 71 to 73% sucrose and 42 DE corn syrup. The sucrose solution is supersaturated partially crystallized. To favor nucleation it is needed to batter (in fondant cannon) the preparation.

A molecular approach of the phase rearrangements of sucrose in presence of anti-crystallizing agents is made. The effect of additives on sucrose nucleation is analyzed. Hydration of sucrose and the formation and stability of sucrose clusters is described. The mechanism of formation of sucrose hydrates seems related to the stability of hydrated sucrose clusters at sub-zero temperatures.

Among the different parameters leading to the instability of sucrose hydrates, the mobility of water in the concentrated amorphous solution seems important. Different rearrangements occur as frozen material temperature is raised. When the temperature reaches the so-called ante-melting (AM) transition, water mobility increases and leads to the collapse of organized sucrose clusters and sucrose hydrates. The bloom is then dissolved to give holes or craters at the surface of icing. Mobility of water is limited by the addition of anti-crystallizing additives which increase the viscosity of the preparation.

The formulation of fondants and the control of size distribution of the crystalline phase is important. The choice of anti-crystallizing additive for its viscosity enhancer and humectants properties is recommended. Analysis of the stability of hydrated sucrose clusters shows the preponderance of the role of water mobility in this type of products.

Theoretical aspects of water-holding in meat

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As myofibrils consist of a three-dimensional network of long, solid protein particles with shortest dimension of less than 20 nm, the theoretical foundations of water-holding in meat should be studied from a colloid or surface chemistry point of view. The classical hypotheses for water-holding in meat are based on electrostatic forces or osmotic forces, which cause the swelling of the myofibrillar lattice. Short range surface forces seem to dominate theories of water-protein interactions, but the theoretical foundations of bulk water holding are still lacking. Our research adds to this the structure of water, i.e. low density water or high density water induced by kosmotropic effects or by chaotropes, respectively. The interactions of ions and non-polar kosmotropes with water and proteins have a relevant effect on water-holding.

The chaotropic/kosmotropic effects of different ions will be of importance especially when reducing sodium contents in meat-based foods. We provide a hypothesis of the chloride ion binding on the hydrophobic surfaces of the core of myosin filaments. This will induce a negative charge to the filaments which will explain the well-known increase of negative net charge of myofibrillar proteins and thus give basis to the theories of water-holding in meat. The binding of the chaotropic chloride ions will also increase of the amount of low density water which will mean an improved water-holding.

Our calculations of the surface areas of major constituents of the myofibrils showed that transverse elements have larger contact surfaces with the liquid phase than longitudinal. Therefore, more attention should be paid to heavy meromyosin, Z-line and other elements of molecular size or colloidal size. Irrespective of the lack of theoretical explanation on the mechanism of water-holding in meat, the meat industry, however, is able to control the macroscopic behaviour of meat-based ingredients rather well.

Metrologically robust moisture measurements

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The measurement of moisture in food, and generally, involves numerous techniques. There are well-known complications of how techniques distinguish water from moisture, and free water from water at various levels of physical and chemical binding. In some cases, results are method-dependent. Calibration and reference methods also suffer from many of these problems, and certified reference materials available for moisture are of limited material types and applicability. Furthermore, the correct scaling of other measured properties of foods is affected by the ability to correctly evaluate dry mass, wherever measurements are referred to this as a baseline. To be metrologically robust, measurements must address these points with due allowance for measurement traceability and uncertainty.

Some work to address these issues will be presented in the form of several examples. Measurements of moisture and (specifically) water content have been made on samples including oats, rice powder, freeze-dried berries, and cellulose. Advantages are gained wherever two different techniques can be used in combination either simultaneously or separately. Examples will be shown of mass loss on drying combined with water-specific coulometric evolved-vapor analysis (to measure both water and moisture content), and microwave resonance combined with humidity conditioning (to measure real-time changes in sample water content).

Example results will be presented with an emphasis on developing metrologically sound measurement approaches, with a focus on measurement traceability and rigorous analysis of uncertainty. The presentation will include an example detailed uncertainty analysis.

New developments of online NIR measurement in food processes

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Online NIR measurement started about 40 years ago with a two-wavelength filter wheel instrument to measure moisture in food. In food production, the constituent moisture is one of the most important parameter especially when drying processes are included. In the following years analogue systems with two-reference wavelengths were developed.

The next big step in upgrading the NIR systems came in the eighties. Customers were looking for online systems to control the drying process i.e. easier calibration, using a microprocessor along with a memory unit for many calibration curves. New parameters for the food industry with the respective filters and algorithms were developed to measure fat, oil, caffeine, protein and alcohol.

One manufacturer increased the rotation rate of the filter wheel up to 7500 rpm. This very high speed produces a full measurement at a rate of 7.5 ms. The increase in measurements per second leads to a much better accuracy of the measured value. Four constituents can be measured at this speed, simultaneously.

In the last years, new systems entered the market. One principle is using an AOTF (Acousto Optic Tunable Filter) for producing the desired spectrum, which falls on a diode array for evaluation. Other systems are based on grating systems for producing the full spectrum and evaluate it by a diode array. Both systems have no moving parts. In order to reduce the calculation period certain data reduction techniques are used like PLS (partial least square) and PCA (principle component analysis).

The online spectroscopy has now reached the level that more and more companies who are manufactures of process machinery have started to integrate NIR gauges to control the process. Research people see the future to use online spectroscopy as so called “finger print” to identify any irregularity in the food.

Karl Fischer water titration of cyclodextrins: parameter optimization

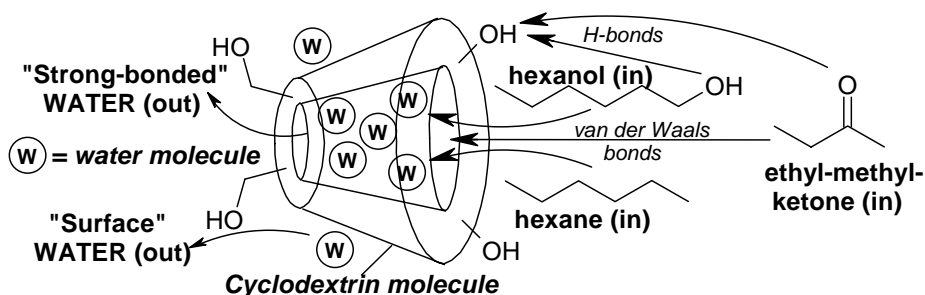
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Water naturally occurs in cyclodextrins (CDs), as well as in cyclodextrin complexes. The water concentration in cyclodextrins and their complexes is very important due to the fact that it is replaced by biologically active molecules in the molecular encapsulation process. Furthermore, a low water concentration in complexes, especially “strong-bonded water” from CD cavity, indicates a good biocompound-CD encapsulation/interaction.



In this study the optimization of the Karl Fischer titration (KFT) method parameters was performed for natural (α , β , and γ CD) and semi-synthetically modified cyclodextrins (hydroxypropyl- β CD). Among other parameters used for optimization (such as temperature, sample mass, and particle dimensions), the hydrophobicity of the solvent was the most important. Three classes of organic solvents were used in order to modify the hydrophobicity of the component 2 from the two-component KFT method: C₂–C₈ alcohols, C₅–C₁₂ alkanes, and di-alkyl-ketones. According to octanol-water partition coefficient of the solvent mixtures, the water content of the studied cyclodextrins varies in a range up to 1.5% (i.e. from 14.6% to 16.1% in the case of β CD in methanol and methanol-butanol systems, respectively). Similar results were obtained also by using alkane and ketone-containing solvent mixtures. Furthermore, the rate of water reaction in KFT analysis reveals the “surface” and “strong-bonded” water in cyclodextrin samples.

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Traceability of water activity measurements

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Traceability of water activity measurement has a large effect on scientific research and trade of food products. When using measurement results in global trade or international scientific community, it is essential that the applied measurement units are of the same size and the uncertainties associated with the results are known and realistic.

As far as the water activity is concerned, measurement results should be linked to the definitions of SI-base units metre, kilogram and kelvin through primary realizations of humidity quantities. An unbroken chain of calibrations and appropriate methods for determining uncertainties are needed to obtain the traceability. Reference materials have usually significant role for most operators in establishing the traceability.

This paper describes possibilities to establish traceability in water activity measurements. Contributions of various error sources to the calibration uncertainty are studied and different calibration schemes are discussed.

a_w of amorphous food solids: Equilibrated versus apparent values

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Water activity (a_w) of solid amorphous food products is a key parameter that governs long term physical stability and moisture transfer within products. By definition a_w represents an intrinsic property of the material that should be measured at equilibrium. Nevertheless, most of the usual methods to obtain a_w consist in measuring it in the head space of the sample, either through capacitance sensors or dew point measurements. As consequence, an accurate a_w implies that surface and core of sample particles are fully equilibrated. In reality, many of amorphous food solids products result from processes such as spray-drying, freeze-drying, extrusion, milling, or dry mixing that induce gradient of water content within the particles after manufacturing. In this case, only an apparent a_w can be measure as it will finally correspond to the surface of the particles and not the whole particles. A key mechanism to take into account when considering such non-equilibrium situation (i.e. water content gradient) is the diffusion of water within the matrix. It is well known that this diffusion strongly depends on the composition, the structure, and the temperature, and can take very long time, especially in dense glassy materials.

The aim of this work is to demonstrate that using an appropriated thermal treatment inside the closed a_w measurement cells allows eliminating gradients of water content within particles. This heat shock should be controlled in terms of temperature and duration to avoid any phase transitions or degradations. With this thermal treatment, it is finally possible to measure the equilibrated a_w that may significantly differ from the apparent a_w usually measured. Such thermal treatment while keeping moisture content constant can be achieved with the new Decagon Aqualab 4TE device up to 60 °C. Few examples on various matrices will be used to illustrate the benefit of this approach to obtain equilibrated a_w . In addition, the impact of short time exposition to lab environment on a_w measurements will also be demonstrated. Finally, it will be also shown that the new Aqualab device also allows establishing equilibrated a_w at various temperatures in the range of 15 °C to 60 °C.

Phase changes observed using dielectric spectroscopy while scanning either temperature or water activity

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It is generally accepted that phase changes, characterized by abrupt changes in molecular mobility, are influenced by both temperature and moisture. Traditional methods for investigating phase transitions have focused on thermal techniques while holding moisture constant. Examples of these methods include thermal mechanical analysis and differential scanning calorimetry. These tests determine critical temperatures for phase changes such as a glass transition temperature (T_g). Recent developments in the generation of high resolution dynamic moisture sorption isotherms has made it possible to observe phase changes by scanning water activity while holding temperature constant. However, instruments that can detect phase changes while scanning either temperature or moisture have not been available. The objective of this study was to develop a technique using dielectric impedance spectroscopy that can identify a phase change when scanning either temperature or water activity. Dielectric spectroscopy has the advantage of measuring sample properties that are not directly dependent on changes in thermal or sorption properties to indicate a phase change. Dielectric properties have previously been used to investigate phase changes through thermal dielectric analysis, but not through moisture-based analyses. To determine if a phase change could be observed by tracking changes in dielectric properties when scanning either temperature or water activity, an instrument containing a specially designed dielectric sample cup housed in a temperature and humidity controlled chamber was designed. This instrument made it possible to hold temperature constant while scanning water activity or sample water activity constant while scanning temperature. Polydextrose was analyzed and a phase change was identified by the loss of a shoulder peak in the phase angle spectrum. This shoulder peak loss occurred at the same water activity and temperature when scanning either temperature or water activity. The results also agreed well with T_g data from the literature and indicate that phase changes induced by temperature or moisture are equivalent.

How water activity can reduce food waste and improve product quality

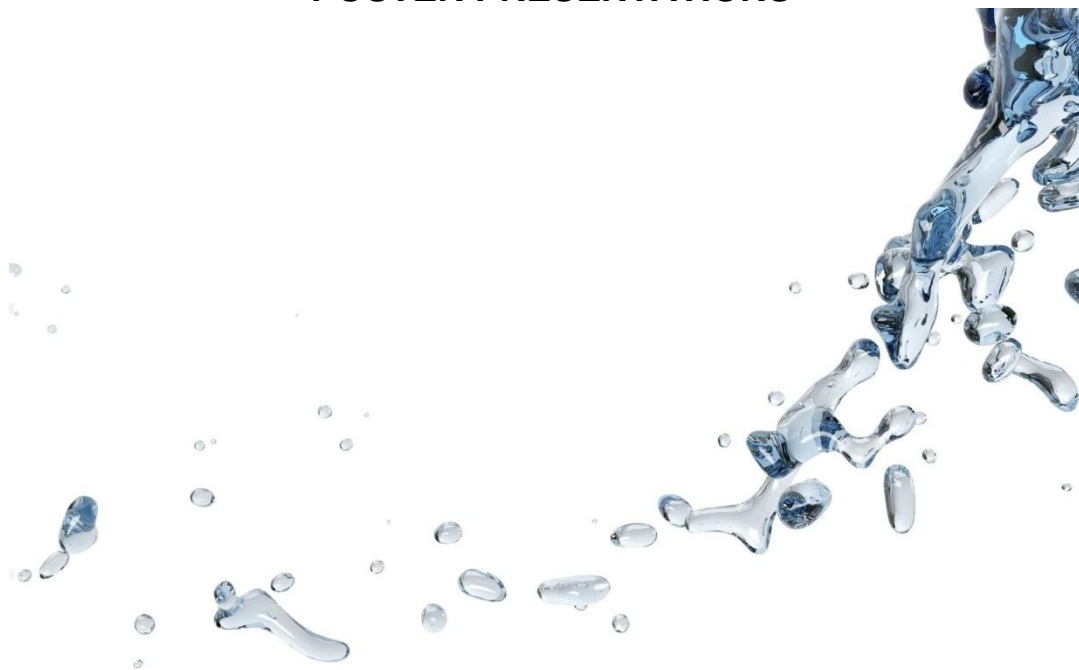
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Several reports and newspapers have recently published that one third of food produced for human consumption is lost or wasted globally. One of the major issues is shelf-life and product stability. In most of the cases no or just little food analysis is done but with just one additional measurement of water activity, more precise proposition can be given.

The measurement of water activity can help the manufacturer to improve the food quality and gather information about the risk of microbial spoilage if his food products. Furthermore, it offers the opportunity to introduce corrective measures to improve the biological, chemical and physical stability of a product and finally get a higher level of food safety and quality plus less food waste in the end.

What water activity is and what powerful opportunities it offers is discussed in this presentation.

EFW 2012
POSTER PRESENTATIONS



Session I

Water and Physical State of Food

Influence of bound and free water on the dielectric properties of extruded starch based pellets

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The production of third generation snacks has increased marketability in the last two decades. These snacks are produced in two steps: First an extrusion process is conducted without expansion of the product. Afterwards in the second step this “half product” is expanded by hot air, deep fat frying or microwave application. In this way both process steps, extrusion and expansion, can be conducted in a separated, for the final product optimized way.

Regarding a microwave induced expansion step, the dielectric properties of the “half product” are very important. They are strongly influenced by amount and type of the ingredients, the product temperature during the expansion step and the water content respectively the a_w -value.

In this study the influence of water content and porosity of extruded starch based “half products” on the dielectric properties were investigated. The “half product” contained wheat flour, 10% sugar and up to 38% water. The corresponding desorption isotherm was measured at 25 °C and the values were fitted with a BET isotherm. The dielectric properties were measured by a network analyzer equipped with a cylindrical resonant cavity.

By comparison of the desorption isotherm with the dielectric properties we can suggest that up to an a_w -value of 0.7, in the BET region, only weak interactions between the bound water and the applied electromagnetic energy are possible. It seems that most interactions result from the starch based matrix. Only at a_w values higher than 0.7, when the region of capillary condensation is reached, the dielectric properties increase dramatically. We conclude that only with free water significant interactions with the applied electromagnetic energy are possible.

Effect of different hydration profiles on physical and mechanical properties of rye bran extrudates

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Water has a significant role in expansion of extruded snacks. The pressure difference between the atmospheric pressure and the vapor pressure of water results in expansion during extrusion. Water also is crucial for starch gelatinization. Higher amount of gelatinized starch gives better expansion rates (Jin *et al.*, 2005). Extrusion cooking is a short time, high temperature and high shear process which limits time for water absorption by the raw material and in turn the food matrix. Changes in hydration profiles prior to extrusion will allow adequate time for the water absorption of starch in the rye bran extrudates.

Rye bran with three different particle sizes (coarse: 450 µm, medium: 150 µm and fine: 24 µm) were processed with a co-rotating twin-screw extruder Thermo Prism PTW-24 (Thermo Haake PolyLab System, Germany) using two screw speeds (300 and 500 rpm), two temperature profiles (sections 1-6 and die: 40-70-75-75-90-110-110 °C and 40-70-75-90-95-130-130 °C) and three different hydration profiles (in barrel moisture contents 17 and 19% and preconditioned 17%). The properties of the extrudates including expansion ratio, density, water absorption index (WAI), water solubility index (WSI), water retention after 120 min (WR120) and textural hardness were evaluated.

Particle size of bran and extrusion conditions affected all the physicochemical properties. The influence of preconditioning was more pronounced with the decrease in particle size compared to varying extrusion operating conditions (screw speed, temperature). The highest expansion rates were achieved by the combination of fine particle size, low moisture content and high screw speed. The textural hardness values were found to decrease with the reduction of moisture content. Extrusion increased the WAI and WSI as compared to the raw material.

Effect of oat bran concentrate on the properties of extruded snacks

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Oats are not commonly used in foods although they contain a lot of dietary fibre particularly β -glucan, which is proved to have health effects. The high lipid content of oats makes it difficult to use in extrusion; lipids are reported to decrease the expansion of extrudates. The aim of this study was to investigate if expanded snacks containing defatted oat bran concentrate could be made by extrusion. Moreover, effects of process variables on the properties of the extruded snacks were studied.

A co-rotating twin screw extruder was used to prepare extrudates containing defatted oat bran concentrate (10% of solids) with defatted oat endosperm flour and with conventional corn flour. In addition, defatted oat bran concentrate (20% of solids) was used with a mixture of defatted oat endosperm flour and waxy corn starch. Physical properties (water content, expansion and hardness) of the extrudates were determined.

Expanded snacks containing defatted oat bran concentrate could be made by extrusion. Extrudates containing defatted oat bran concentrate with corn flour had higher expansion and smaller hardness than extrudates with defatted oat endosperm flour. Due to addition of waxy corn starch higher amount of defatted oat bran concentrate could be used with defatted oat endosperm flour in order to obtain well expanded snacks. However, process variables affected the properties of extrudates: increasing screw speed and decreasing water content of the mass increased expansion and decreased hardness of the extrudates.

A new process for a traditional apple-derived product obtained using an innovative sustainable solar drying system

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In the last few years consumers are becoming more interested in healthy, natural and tasty foods, and dried food products represent an important market area of innovative snacks, in particular fruit and vegetables based products. However removal of large quantities of water from fruit implies the use of huge amounts of energy.

Exploiting renewable energy sources seems to be a winning path in a sustainable production context and besides the economical aspect, development and application of energy saving systems can also involve significant quality improvement to the obtained products.

Here some quality parameters of apple candies obtained by means of two different techniques were compared: the traditional technique – that involved direct sunlight exposure – and an innovative one, that used a solar dryer system. Kinetics of the two drying processes were monitored during the whole process recording the following parameters: process temperature and humidity, product weight loss, color, texture, and water activity. The final quality evaluation was also performed by a sensory panel test.

Colour and texture data report significant differences between the dehydration techniques: the product obtained applying the innovative system was characterized by brighter color and softer texture than the traditional one, as well evidenced by the panel test results. Samples were monitored over a six months shelf life and data confirmed the quality stability of the final product, validating the innovative solar system.

These results suggested that drying foods by means of solar exposure using a protected system, represents a processing improvement to obtain quality products, especially in small and craft productions.

Structure formation in sugar containing pectin gels – Influence of pH on the gelation of high-methoxylated pectin

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Pectins are branched polysaccharides composed of partial methyl ester polygalacturonic acid and sodium, potassium, calcium and ammonium salts. Pectins are present in all plant cell walls of higher plants and important gelling or thickening agents for the food industry. Traditionally, pectins are applied in jams and jellies, but they are used also in other food products e.g. as stabilisers in acidified milk drinks and as thickeners to improve the texture of oil-in-water emulsions [1].

Detailed knowledge of material and structuring properties, especially the immobilisation of water during the gel formation process, is of crucial importance for product and process design. Depending on the degree of methoxylation, water is immobilized by two main gelation mechanisms during the gelation process. The gelation mechanism of high-methoxylated pectins (HM pectins) is described as a combination of hydrophobic interactions, prevailing at higher temperatures and hydrogen bonds between the undissociated carboxyl groups, dominating at lower temperatures [2]. Low-methoxylated pectins (LM pectins) form gels in presence of Ca^{2+} or other divalent metal ions. Intermolecular ionic junction zones are formed between smooth regions of neighboured chains [3]. As a consequence, the most important factors are pH, soluble solids and divalent ions such as Ca^{2+} .

The aim of the study is to present a method for the characterisation of the HM pectin gel structuring process by using the structuring velocity (dG'/dt) and to apply this method for the investigation of pH influence on the gelation process of HM pectins in the presence of 65% sucrose.

The complex pectin structuring process, the transition from liquid to solid, was determined by oscillation measurements during cooling. The method has been widely used for the examination of the gel point which is the intersection point between the storage modulus G' and the loss modulus G'' . This determination is, however, not always sufficient; for instance in case of pre-gelation often no gel point can be detected. Therefore, apart from the dynamic rheological parameters G' and G'' , the first derivation of G' over time (dG'/dt) as the structuring velocity allows the determination of additional structuring temperatures or phases of the pectin-sugar model systems [4,5]. Moreover, structuring phases can be identified from the curve shape of the structuring velocity during gelation process of HM pectins depending on acid content, as well as an optimum range of the final gel structure at tartaric acid content of 22.3 mM/kg gel.

The additional structuring temperatures have proved to be valuable parameters of the incipient pectin gel structuring process, and can help to understand the change of state from liquid to solid under varying gel compositions during cooling.

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Role of water as plasticizer of novel wood-derived edible films

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Spruce (*Picea abies*) galactoglucomannans (GGM) are polysaccharides that can be recovered at high yield and low cost as forestry industry by-products: from thermomechanical pulping process water by purification and ultrafiltration following alcohol precipitation or spray-drying, from wastewater of fiberboard mills by ultrafiltration, or from wood chips by pressurized hot-water extraction. GGM are a new potential natural raw material, but they are not yet isolated or used in an industrial scale. We have recently studied the properties of GGM as edible and biodegradable food packaging film formers. Other potential uses for GGM could be as emulsion stabilizers or microcapsules. Since water acts as plasticizer of polysaccharides, we characterized the properties of GGM films as a function of relative humidity (RH). Dynamic vapor sorption, water vapor permeability (WVP), and moisture-scanning dynamic mechanical analyses were performed. Increasing RH resulted in a decrease in the storage modulus of the films, i.e., the films softened due to moisture-induced glass transition. Increasing amount of added polyol plasticizer decreased the RH of softening of GGM films and increased the moisture uptake of films at high RH. In addition, the WVP of films increased with increasing RH. Spruce GGM are potential novel food hydrocolloids and edible film formers, but the moisture sensitivity of GGM films should be addressed, e.g., by crosslinking or lamination of the films with a hydrophobic layer.

Multiple protein glass transitions at low water content

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When concentrated protein solutions enter the glassy state their physical transport properties change sharply, making these systems potential candidates as e.g. coating material. While their macroscopic physical properties have been widely examined, the underlying microscopic physical mechanism it is not well understood. This project focusses on the properties of β -lactoglobulin (β -lac) solution near the glassy state. The dynamic behaviour of high-density protein systems are examined on multiple length scales using several techniques.

Using micro-rheology (Diffusion Wave Spectroscopy), no sudden increase in the decorrelation time (\sim inversely proportional to the viscosity) was observed between 20–80% protein content. This suggests no sharp glass transition is present at the typical length scale probed (600 nm). The next step is to compare these results with glass transition temperatures at different protein contents as determined by differential scanning calorimetry and local thermal analysis. In addition, thin layer MRI is used as an additional technique to determine water mobility and distribution in coatings of high protein content.

Measuring water vapor permeation for food package materials using tritium

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Many packaged foods require a barrier to gases, flavors or odors to maintain product quality and provide acceptable shelf life. And various barrier coated polymer films are being used to package many different food products. A continuing trend of food packaging is to design to extend the shelf life of food while maintaining fresh-like quality. So the specific moisture permeability is critical factors in selection of food packaging materials. This study established a method for measuring a water vapor permeation in a range ($10^2 \sim 10^{-6}$) [$\text{g}/\text{m}^2\text{day}$] by using water including tritium that is a radioactive isotope. This method provides absolute quantitative measurement for water vapor permeation. An uncertainty of a manufactured measurement device was about 1×10^{-6} [$\text{g}/\text{m}^2\text{day}$]

Session II

Water and Stability of Food

Water-induced plasticization of spray-dried maltodextrin-caseinate matrices

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The recent progress in understanding plasticization of glassy carbohydrate matrices at a molecular level provides useful setup for attempts to correlate structure and structural changes with function. The approach of using glassy carbohydrates has been a generally accepted strategy for designing optimal encapsulation systems. However, the recent observations indicate that besides glass transition temperature also the nano-scale structure of carbohydrates control the molecular mobility of small permeants (like oxygen) in glassy carbohydrate matrices. In this study water-induced plasticization of spray-dried maltodextrin-caseinate matrices (non-cross-linked and TG cross-linked caseinate) was studied with DVS, DSC and low-field ¹H-NMR after equilibrating the powders at constant relative humidities (0, 11, 33, 54 and 75%).

Surprisingly, caseinate cross-linking was found to reduce matrix water vapour in glassy state in DVS measurements. However, a long-term water vapour sorption experiments in humidity chambers showed that in true equilibrium there was no difference between the sorption isotherms. Thus, cross-linking of caseinate slowed significantly water vapour diffusion rate in glassy matrix.

Glass transition temperatures were measured for maltodextrin with DSC and ¹H-NMR and a good correlation was found for the two methods. The samples stored at RH 54% and below that were in glassy state, whereas the structurally collapsed powder at RH 75% was in rubbery state. Further on, the observed reduction in M₂ parameter (measure of molecular mobility) from ¹H-NMR measurements in glassy state could indicate molecular scale swelling of the maltodextrin matrix.

Water migration mechanisms and agglomeration propensity in amorphous powder material varying in composition, particle size and morphology

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The agglomeration phenomenon of amorphous particulate material is a major problem in the food industry. Insights in the kinetics and mechanisms of water transport in powder material can help to better understand and control powder agglomeration. For this purpose, maltodextrins (MDs) varying in dextrose equivalents (DE), particle size and morphology were investigated. Variation in particle size and morphology were obtained by producing powders with conventional spray dryers as well as with an ink-jet technology developed by TNO. The ink-jet technology results in mono dispersed spherical particles in contrast to the variety in shapes and morphologies from conventional spray dryers. Gravimetric step-change water sorption experiments were performed on the MDs as a function of the a_w . The experimental results were compared with a Fickian diffusion model in order to understand the dependency of the transport mechanism on water concentration gradient and material relaxation. The water transport kinetics in the MDs with low DE (i.e. 6) were well described by Fickian diffusion for low a_w , independently of particle size and morphology, until relaxation phenomena started to occur at an a_w corresponding to $T - T_g = -20$ °C. The importance of the matrix relaxation phenomena on the water transport mechanism increased with increasing DE (i.e. 29 and 32), not showing any relationship with the T_g . The results of this study indicate that the water migration mechanism is controlled by relaxation phenomena when the amorphous material is still far from the glass-rubber transition. The $T - T_g$ at which the relaxation phenomena occur depends on the material. On the contrary, the $T - T_g$ parameter could well describe the onset of agglomeration, independently from the material properties.

Effect of relative humidity on stability of bulk and encapsulated sunflower oil

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Lipid oxidation is the major reaction leading to deterioration of foods containing unsaturated oils. Chemical composition and physical factors, including presence of water, have an important role in the stability. Depending on the continuity of the lipid phase, effects of water may vary. In order to produce stable foods with highly unsaturated oils, there is a need for structures that limit the contact between lipids and oxygen. Encapsulation by spray-drying of oil-in-water emulsions is one possible approach to reach this goal. In this study, the effect of relative humidity (RH) on the stability of bulk and encapsulated sunflower oil was studied.

Sunflower oil was encapsulated in a maltodextrin-sodium caseinate matrix by spray-drying. The bulk and encapsulated sunflower oils were stored under different RH (~0, 11, 33, 54, and 75%) at 22 °C in the dark for 29 weeks, and studied at certain time points for peroxide values (PV) and residual α -tocopherol.

RH had only a minor effect of the stability of sunflower oil in bulk. The PV of the oil increased to 100–130 meq/kg and the α -tocopherol content decreased from 490 to ~370 μ g/g after 29 weeks under all RH. However, RH had a greater effect on the stability of the encapsulated oil. The highest PV and greatest losses of α -tocopherol were obtained at RH ~0% (PV 140 meq/kg, residual α -tocopherol 210 μ g/g) followed by RH 11% and 33%. Oxidation of the encapsulated oil started immediately at these low RH. Interestingly, encapsulation seemed to protect the oil when stored at RH 75%, since the PV increased only to 20 meq/kg. This was obviously due to the collapse of the matrix by high humidity. The oxidation behaviour of the encapsulated oil was thus considerably influenced by the RH in comparison to the bulk oil.

The use of front face fluorescence spectroscopy for measurement of lipid oxidation during storage of cakes at 20 °C and 65% of RH

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Consumers demand foodstuffs with superior quality as well as minimally processed food retaining the fresh products' features. During storage, food products are subject to changes, resulting in adverse effects on quality, ranging from minor organoleptic defects to total spoilage. Nowadays, bakery products are widely consumed and therefore particular requirements for their quality characteristics during storage have been established. Lipid oxidation is one of the most common causes of cake alterations occurring throughout the storage constituting the major concern for food industries. Traditional physico-chemical analyses determining the level of primary and secondary products throughout storage are usually used. However, these methods are destructive, need skilled operators and the use of pollutant reagents. In the present study, front face fluorescence spectroscopy (FFFS) coupled with chemometric tools was used as a useful tool for the monitoring of sponge cakes freshness, produced at the pilot scale, during ageing. The fluorescence emission spectra were acquired in the 340–490 nm and 390–680 nm after excitation sets at 325 and 380 nm, respectively while excitation spectra (250–390 nm) were scanned after emission set at 410 nm. The principal component analysis applied separately to each spectral collection allowed a clear discrimination of cakes according to their ageing (i.e., 1, 3, 6, 9, 16 and 20 days). These results showed that FFF spectra provide useful fingerprints, mainly allowing the identification of cakes during storage and could be considered as a powerful tool for the evaluation of cake freshness.

Keywords: Sponge cakes, Freshness, Oxidation, Fluorescence, Chemometric

Use of amaranth, quinoa and kañiwa in extruded corn snacks: Effect of humidity exposure on lipid oxidation

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There has been a growing concern about the prevalence of gastrointestinal disorders associated with celiac disease and gluten sensitivity. Amaranth, quinoa and kañiwa are pseudocereals cultivated in South America that could become attractive food alternatives in Europe due to their status as gluten-free, protein quality and unsaturated fatty acids. The aims of this investigation were to prepare corn-based snacks containing amaranth, quinoa and kañiwa using extrusion cooking and to evaluate lipid stability of snacks exposed to different humidity conditions (11% RH, 76% RH). Extrudates were made during 4 separate extrusion trials using a co-rotating twin screw extruder. Amaranth/quinoa/kañiwa (20% of solids) was mixed with corn flour. Pure corn flour was used as control. Experiments were performed using Box Behnken's experimental design, in which extrusion process variables were water content of the mass (15, 17 and 19%), screw speed (200, 350 and 500 rpm) and temperature of the die (150, 160 and 170 °C). The physical properties of snacks such as Sectional expansion index (SEI) and hardness were determined. Extrudate samples (ground and whole) were stored in open headspace vials at 11 and 76% RH for a week before being sealed and stored for 9 weeks in the absence of light. Formation of hexanal was evaluated using headspace gas chromatography at intervals during storage.

Extrudates containing amaranth presented the highest sectional expansion index (SEI) compared to other extrudates. In contrast, pure corn extrudates (control) exhibited the lowest SEI and highest degree of hardness. Whole extrudates exposed to 11 and 76% RH showed lower hexanal production than ground extrudates. Indeed, whole extrudates exposed to 76% RH presented the lowest and most constant hexanal production during storage. This study showed that amaranth, quinoa and kañiwa can be successfully combined with corn flour leading to expanded extruded snacks; and that exposure to high relative humidity (prior to storage) might retard lipid oxidation in whole extrudates.

Multianalytical approach to study water state, mobility and antiplasticization effects on coffee beans

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The approach of combining different techniques to investigate the status of water in food systems has been recently adopted. Sorption isotherms, thermal analysis, such as differential scanning calorimetry (DSC), LF-NMR offer different but complementary points of view in the study of the dynamics of the water binding process as well as the water status. Recently the dielectric properties received a renewed interest as they are influenced by several matrix and environmental factors (composition, structure, density, temperature, frequency of the applied wave) as well as moisture content and a_w . This study reviews the toughening and plasticization effects of water on coffee beans in terms of texture modifications and water status and mobility, investigated by the joint use of sorption isotherms, DSC, LF-NMR and integrates the results of the dielectric properties assessment currently ongoing on green and roasted coffee beans. This multi-analytical approach is aimed to a better understand the role of water status in the determination of the toughening and plasticization effect. From BET monolayer value to $a_w = 0.61$ and 0.75 for green and roasted coffee, respectively, the solid matrix hydration occurred and water induced hardening. Very short NMR T_2 values and the concomitant absence of any DSC endothermic peak assignable to water freezing were observed at these a_w values. When solid matrix hydration was completed, water started to act as a plasticizing agent, NMR revealed the appearance of a new proton pool with increased mobility and by DSC peaks associated to water freezing were determined. Above this hydration degree ($a_w = 0.78$ and 0.86 for green and roasted coffee, respectively), water determined a decrease of bean hardness and a further decrease of the elastic modulus. The dielectric properties characterization of coffee beans at the different hydration levels will integrate the knowledge of water state and mobility in coffee matrix, and its relation with textural aspects.

Microstructural and textural properties of frozen carrots as affected by blanching in water and sugar solutions

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Freezing is a common process used for long term preservation of plant foods. However, irreversible physical changes during frozen storage may compromise the quality characteristics of the product and in particular the texture. The use of sugars could represent a potential action to preserve the quality of frozen vegetables due to their cryoprotective ability of cell membranes and antifreezing effect by the increase of the amount of unfrozen water at low temperatures.

Aim of this study was to study the effects of heating conditions and the sugar type on the mechanical properties and microstructure of the sliced carrots after blanching and during frozen storage. Raw carrots and carrots blanched in water and in 4% trehalose and maltose solutions at 75 °C for 3 (A) and 10 min (C) and at 90 °C for 3 (B) and 10 min (D) were frozen and stored at -18 °C for 8 months.

By scanning electron microscopy (SEM) analysis no significant differences were observed among samples A and B blanched in water and the raw carrot. The thermal damage was prominent in C and D while the thermoprotective effect of the sugars was evidenced only in D. Freezing and frozen storage determined several fractures on raw and blanched carrots due to ice crystals formation. The cryoprotective effect of the sugars was observed only in sample D.

The mechanical properties of carrots were affected by blanching whilst no effect due to the sugar uptake was evidenced. The cryo-protective effect of the sugars during storage in frozen state was observed only in sample B. Microstructural and texture analysis provided different information; the use of both the techniques is thus recommended to gain insight on process-induced modifications.

Physico-chemical properties in stabilized pasta during storage

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The request for convenient food has pushed the pasta food industry to develop ready-to-eat, shelf-stable, high quality pasta products. Such products significantly reduce consumers' acceptance during storage mainly due to texture modification whose fundamental causes are still unknown.

Dry semolina pasta was pre-cooked, packed into retortable pouches (~ 60 g / pouch), sterilized ($F_0 = 7$), and stored at room temperature for 2 months. Textural properties (TA.XT2 Texture Analyzer), and water status at different scales (moisture content, water activity, DSC frozen water content and ¹H NMR mobility [FID, ¹H T₂ distribution, and ¹H self diffusion coefficient D; 20 MHz ¹H NMR, the MiniSpec Bruker]) of the product were measured over the entire storage time.

Pre-cooked stabilized pasta had constant moisture content (~ 57% g H₂O / g sample) and water activity (~ 0.98), while frozen water content was slightly reduced (from ~ 80 to ~ 75% g frozen H₂O / g total H₂O) during the two months of storage. Pasta hardness increased during storage, more importantly in the first 2 weeks of storage. ¹H FID indicated an increased rigidity of the most rigid protons (increasingly steeper FID decay), while the ¹H D was constant during the first 40 days of storage (~ 1.2–1.3 10⁻⁹ m²/s) and then increased to ~ 1.5–1.6 10⁻⁹ m²/s. ¹H T₂ distributions depicted three proton populations (T_{2A} 4 ms, T_{2B} 18 ms, and T_{2C} 40 ms) in the fresh products and, after 3 days, T_{2A} and T_{2B} merged into a single broad population.

¹H NMR mobility changes during storage may suggest that some changes in the water-starch-gluten interactions may contribute to the textural changes in pasta during storage.

Effects of licorice extracts and extrusion process variables on physical properties of licorice

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Licorice is counted to the popular candies in northern Europe, especially in Scandinavia. Even though the strong taste and the black color are typical for licorices, the trend on the confectionery market leads to new flavors, colors and healthier products. Considering those changes, the aim of the study was to determine how different licorice extracts and extrusion process variables affect the licorice. The main focus was in particular on the effects on the water content and the water activity of licorice. Process variables, which were tested, included the feed rate and screw speed of the extruder. Variables in the recipe were content of water and two licorice extracts.

The experiments were conducted according to the 2⁴⁻¹ fractional factorial design, which contained two center points in addition. The licorices were produced with a co-rotating twin screw extruder. The samples differed in the amount of licorice extract (2.5, 3 and 3.5% of solids), the water content in the recipe before extrusion (24, 25 and 26%) and the licorice extract type, which differed in the amount of sugar. In addition to that, the feed rate (60, 75 and 90 g/min) and the screw speed (30 and 40 rpm) were also changed in order to study their effects on physical properties of licorice. Physical properties (water activity, water content, hardness, diameter and storability) of all licorices were determined.

The results of the present study showed that both, the licorice extract and extrusion process variables, affected physical properties of licorice. The changes were observed in the water content, the water activity and the diameter of licorice products. The data obtained can be applied in further development of licorice with desired properties by using the co-rotating twin screw extruder.

Salt reduction in meat processing: Effects on water state and quality of dry cured ham

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Salting is an ancient technology widely used in combination with other actions to preserve meat by water activity depression and development of desired sensorial properties. In recent times the health and nutritional concerns about sodium intake has led to a reduction of the salt content in processed meat products also in the case of the traditional ones. This is not easy task in the dry cured ham as salt plays an important technological role in the control of endogenous enzymes involved in the development of the main quality characteristics and in the water state of the final product.

In this study the effects of changes in the salting process conditions on the salt diffusion and water state of a traditional smoked dry cured ham have been investigated. Raw hams of two different size (small-S and large-L) were subjected to a 2- or 3- salting steps process. Salt content, moisture and a_w were determined on three muscle portions of the ham before and after salting up to 90 days of processing. The decrease of the number of salting steps reduced the salt diffusion and the salt content of the inner regions of the L-hams while no meaningful effects occurred when the same experimental procedure was applied to the small size raw hams. NaCl content and a_w values of all the ham samples resulted to fit a linear model ($r^2 > 0.80$) and the linear equation was used to predict the final a_w value of the differently salted dry cured hams. The predictive model highlighted the risk that based on the salt content determined at the end of the resting, 2-salting steps-L dry-cured hams will achieve an $a_w > 0.92$ value that could impair the microbial stability of the product and compromise the sensory quality.

Session III
Water and Quality of Food

TG–DTA approach for designing reference methods for total solids determination in liquid foods

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Simultaneous Thermo-Gravimetry and Differential Thermal Analysis (TG-DTA) has been described in the previous EFW conference (*Food Chemistry 122, 2010, 436-442*) as a robust method for determining moisture content in most dehydrated amorphous food products (e.g. milk powder, coffee powder, cereal powder and dry pet foods) using the weight loss at the inflection point by heating at a rate of 2 °C/min. It was also shown how to determine the optimal isothermal drying temperature to be applied in the classical oven drying method.

In this presentation, we would like to promote a similar approach based on TG-DTA for determining the total solids in homogeneous liquid foods. By observing the drying profile under constant heating rate (e.g. 2 °C/min), we will focus on the end of the drying, especially when the concentrated liquid goes through the glass transition during the drying to reach the solid state. Using various liquid food model systems, we will interpret the drying pathway as well as the thermal events and propose an approach to identify the end point of the drying and the onset of chemical reactivity or decomposition.

Along the presentation, we will consider the following liquid food model systems:

- Whey protein concentrate as model system for protein rich products
- Glucose syrups (e.g. DE 21) as model system for carbohydrate rich products
- Pet food liquid digest as model systems for lipid rich products

As for dehydrated amorphous food products, this approach will be applied to estimate the optimal isothermal drying temperature for designing robust oven reference methods for total solids determination in most liquid foods.

Water content of wheat flour and bread products by Karl Fischer titration: a PCA multivariate approach

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The water content of cereal products is an important parameter related to the quality of food products during storage. A lower water content than 9% protect the products against infections; a value up to 14% did not conduct to a fungal growth.

In the present study an evaluation of the performance of the determination of water content of wheat flour and bakery products by using the Karl Fischer titration (KFT) method was performed. Three types of wheat flour and four types of bread products were analyzed by KFT and PCA (principal component analysis). The highest content of water was found in the white flour, 14.9–15.2%, while the wholemeal and germ flour have lower water content (14.5–14.7%). In the case of bread from white wheat the water concentration was in the range of 39–43%. An important difference was observed in the case of bread obtained from wholemeal or germ flour (44.1–44.2%) in comparison with the Graham bread (36.5%). In the case of wheat flour, the water reaction rate in the first interval (“surface water”) was in the range of 1.0–2.1 mM/s, while the “strong-bonded” water has a reaction rate of 0.13–0.24 mM/s. Higher water reaction rate in the first interval was obtained for the bread samples (1.7–4.2 mM/s). The “strong-bonded” water appears only for bread made from wholemeal flour or germ flour. All KFT variables were used in the statistical multivariate PCA analysis of all samples; among these, the water content (%) and the normalized titration volume were the most important for a good classification of the flour and bread products. Furthermore, the authenticity of these samples can be established by using this statistical technique.

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Karl Fischer water content of saturated and unsaturated fatty acid / α - and β -cyclodextrin supramolecular systems: Correlation with structural parameters

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All essential fatty acids (especially unsaturated ones) undergo chemical changes (e.g. oxidation and auto-oxidation) even at room temperature and convert to various organic compounds, some of them by means of free radicals with implications in inflammatory and degenerative diseases, cancer, and Alzheimer's disease. The protection against oxidation (and further against generating the free radicals) and controlled release of "good" *cis* fatty acids can be realized by molecular encapsulation, i.e. in cyclodextrins. The quality of encapsulation depends on many factors such as the guest molecule structure and hydrophobicity, encapsulation parameters (temperature, pH, solvents), and finally the water content of the complex can vary according to these factors.

In this study saturated (decanoic, palmitic, stearic, arachidic, behenic) and unsaturated (oleic, linoleic, eicosapentaenoic, and docosahexaenoic) fatty acids / α - and β -cyclodextrin complexes were obtained by controlled crystallisation from ethanol-water and analyzed by FT-IR, DSC, SEM, and Karl Fisher water titration (KFT). The water concentration of α -cyclodextrin complexes was 1.2–3.2% lower than α -cyclodextrin and for β -cyclodextrin complexes this difference was higher (5.2–6.1%). Generally, complexes containing unsaturated fatty acids have higher water content in comparison with the corresponding saturated ones. Two types of water molecules were evaluated: "surface" water, which reacts with a higher rate in the titration process, and "strong bonded" water molecules, which react with a lower rate. These KFT results correlate with the theoretical molecular modeling and docking studies on fatty acid / cyclodextrin complexes.

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Determination and application of sorption isotherms of food

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There is an important relationship between water content and physical, chemical and microbiological properties of foods. Water holding capacity of food is characteristic and specific for different kind of foods because of the heterogeneity, colligative properties, capillarity and surface interactions in the matrix. Alteration in relative humidity of the environment at a constant temperature or alteration in temperature of the environment at a constant humidity affects the food-water relationship. Sorption isotherms are one of the most important methods to explain that relationship including the mentioned interactions. The graph obtained by water content (m) versus water activity (a_w) at a constant temperature is called sorption isotherm, $m=f(a_w)T$. The isotherm is determined by the weight changes between food and its atmosphere till they reach the balance in moisture content in closed spaces that have different relative humidity at a constant temperature. Sorption isotherms are obtained starting from low water activity values for dried foods as adsorption isotherms and from high water activity values for aqueous food as desorption isotherms. It is possible to estimate the critical points by using the sorption isotherms in order to take precautions against the problems such as plasticization, aggregation, losing of crispiness and transition of amorphous structure to crystal structure. If these critical values are taken into consideration during the production and packaging process, foods could be maintained even though marketing is in different areas with different temperature and pressure values. The sorption equations like BET and GAB provide the opportunity to determine the important information like monolayer water content (m_0) of foods which indicates the minimum oxidation level in dry food. Sorption isotherms are also used for selection of packaging material, determining of the shelf life and moisture transfer in mixing ratio of dried ingredients. Conclusion, definition and experimentally determination of sorption isotherms, application of the obtained data to the equations and use of isotherms are important in food industry.

Adsorption isotherms of unsteamed, steamed and honeycoated roasted cashewnuts

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Adsorption isotherms of unsteamed, steamed and honeycoated roasted cashewnuts at 20 °C, 30 °C and 40 °C were determined using a gravimetric- static method. BET and GAB models were applied to the isotherm data. Heats of moisture adsorption were estimated from equilibrium sorption data using the Clausius–Clapeyron equation. Adsorption isotherms of the steamed and unsteamed roasted cashewnuts followed a type II (sigmoidal) shape while the adsorption isotherm for the honey coated roasted cashewnut followed a type III (J-shaped) shape. The adsorption isotherms were best described by GAB equation. Equilibrium moisture content decreased with increased temperature at low and intermediate moisture contents for both unsteamed and steamed roasted cashew nuts. Inversion of the effect of temperature on adsorption isotherms of honey-coated roasted cashewnuts occurred at $a_w > 0.64$, showing the endothermic dissolution of sugar at high water activities. Calculated isosteric heats of sorption increased with decreasing moisture content, showing that the strength of interactions between water vapour and the adsorbent increased with lower moisture content. Negative isosteric heat of sorption occurred only in honey-coated cashew nut at higher moisture (>0.24 g water/g dry solids) contents.

Adsorption isotherms of fresh and osmo-oven dried bananas and plantain slices

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Adsorption isotherms of fresh and osmotically pretreated and subsequently oven-dried banana (Omini Red, Cavendish and Cooking banana) varieties and plantain were investigated at 20 and 40 °C using gravimetric static method. Firm ripe (stage 5) plantain and bananas were transversely cut into 10 mm slices and osmotically pretreated in 52°, 60° and 68°B sucrose solutions for 12 h at 25 °C. Both fresh and preosmosed plantain and banana slices were subsequently oven-dried at 60 °C for 72 hours. Suitability of eight sorption models in describing the adsorption data was tested. Moreover, isosteric heat of sorption of both fresh and preosmosed oven-dried bananas was determined using Clausius–Clapeyron equation. Adsorption isotherms of fresh and preosmosed oven-dried banana and plantain slices gave type III (J-shaped) isotherms. Isotherms were affected by temperature and pretreatment sucrose solution concentration. Crossing of isotherms occurred at $a_w \sim 0.55\text{--}0.70$ and $a_w \sim 0.60\text{--}0.70$ for bananas and plantain, respectively. Among the eight models tested, Guggenheim-Anderson-de Boer (GAB) model gave the best fit. Calculated GAB monolayer moisture (M_m) content (9.80–20.8% d.b.) decreased with increase in temperature. Generally GAB M_m content increased with increase in total solids (%) content of preosmosed banana slices. Isosteric heat of sorption increased with decreased moisture content but increased with increase in sucrose solution concentration.

Blood pressure lowering peptides from wheat gluten

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High blood pressure or hypertension is an important worldwide health problem because of its high frequency and associated risks of cardiovascular and kidney diseases. Renin-angiotensin system (RAS), which mediates vasoconstriction and extracellular volume, is the most important system that regulating blood pressure. ACE inhibitors in RAS can lower blood pressure. Hypertension drugs, like captopril, are usually ACE inhibitors. However, long-time take of hypertension drugs causes dry cough and other severe implications. ACE-inhibitory peptides can be obtained from various food protein sources. Applying ACE-inhibitory peptides in food matrix and developing food products containing high content of bioactive peptides are desired. Wheat gluten, a co-product from starch industry, is a potential source for obtaining ACE-inhibitory peptides.

This work studied substrate wheat gluten degradation by enzymatic hydrolysis of thermolysin or/and prolyl endoprotease from *Aspergillus niger* (AN-PEP, Clarex). The hydrolysis condition, like incubation time and substrate to enzyme ratio, was determined by SDS-PAGE, free amino nitrogen content and size-exclusion chromatography. Followed by ultrafiltration with 3kDa cut-off after hydrolysis, the *in vitro* ACE-inhibition activities of hydrolysates was measured, and expressed by IC_{50} value. Furthermore, purification of hydrolysate was carried out by reverse phase liquid chromatography, and coupled with mass spectrometry targeted ACE-inhibitory tripeptides was identified.

The optimum condition for thermolysin hydrolysis of wheat gluten was pH 8.0, at 40 °C for 4 hours incubation, and for Clarex hydrolysis was pH 4.0, at 70 °C for 22 hours. The sample hydrolysed by thermolysin first and then, by Clarex contained 126.6 mg/l free amino nitrogen, and main molecular distribution was under 6.5 kDa. After 3 kDa cut-off ultrafiltration, first thermolysin then Clarex treated hydrolysate showed strongest ACE-inhibition activity, IC_{50} value 0.016 mg protein/ml. According to an external standard, ACE-inhibitory tripeptide Leu-Gln-Pro (LQP) was identified. Targeted liberation of ACE-inhibitory peptides by specific enzymes and isolation by membrane filtration techniques appears a promising approach to produce blood pressure lowering peptides from wheat gluten.

Impact of steam cooking process on moisture, aroma and heat transfers

Descours E.¹, Ferret E.², Debeaufort F.¹, Voilley A.¹ and Seuvre A.-M.¹

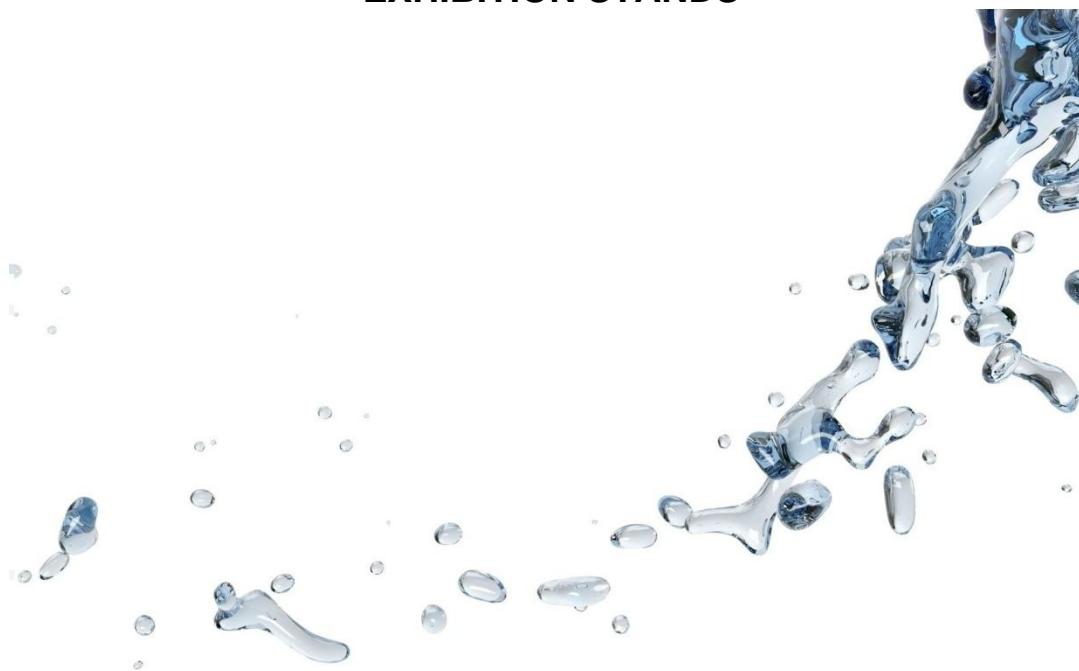
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Recently, the number of diseases due to an unbalanced diet has increased. Therefore, the right choice of cooking preparation can help in overcoming the health problems. The most promising method that preserves nutritional as well as organoleptic food properties, seems to be steam cooking.

The aim of this study is to model heat, moisture and aroma transfers during cooking process in a steam cooker to understand steam cooking process impact on the products qualities. Study has been conducted on potatoes because it's most commonly steam cooked product. Mass transfers were determined both for water and for hexanal as one of the most abundant aroma compounds found in potatoes. Moreover, other water vapour based heat treatments (at 120 °C and 140 °C) and one treatment combining infrared and water vapour were done. The modelling and experimental results were compared. Heat transfer and mass transfer characterizations were performed using the simulation software COMSOL on a potato assimilated to a cylinder cooked in a steam cooker. During the steam cooking, the cooking chamber is progressively saturated with the water vapour. Therefore, water molecules tend to diffuse in the potatoes placed in the cooking chamber. In raw potatoes, aroma compounds like hexanal are preferentially present in the central part. During cooking process, as the water content in the potato increased, the concentration of the hexanal situated in the periphery part of potato matrix was preferentially lost. It was attributed to its diffusion towards external atmosphere. The infrared processing does not influence the diffusion of water and hexanal. Nevertheless, it allowed the development of aromatic compounds due to the Maillard reactions that do not appear in the conventional steam cooking. The cooking time was significantly reduced with the cooking temperature increase.

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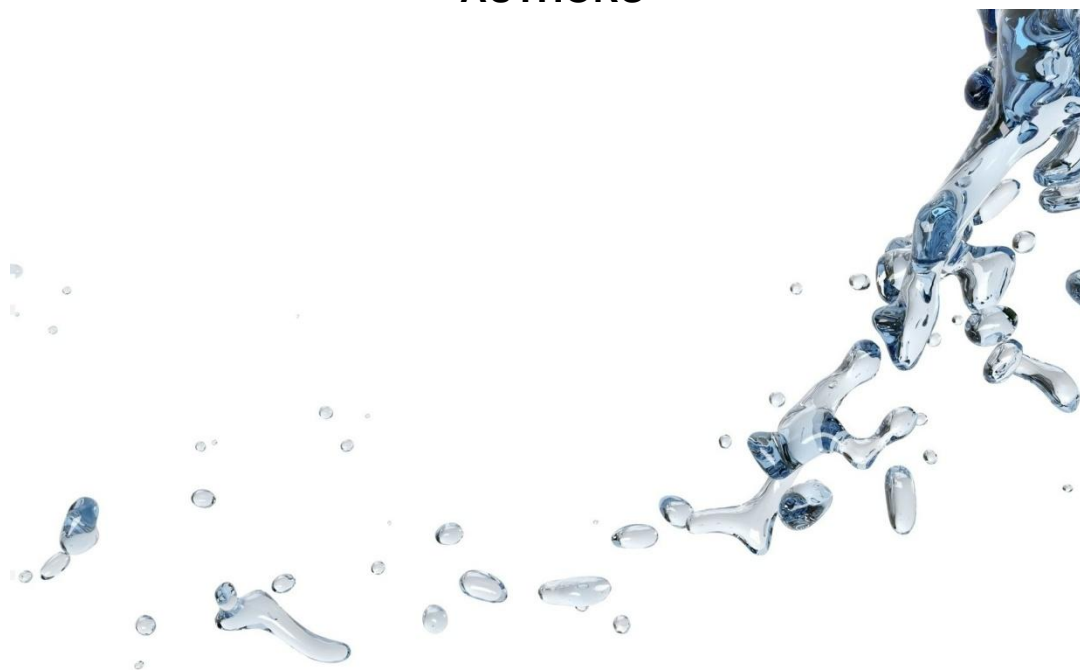
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