



Association EuroFoodWater

# ***BOOK OF ABSTRACTS***

**8<sup>th</sup> International Conference on**

## **WATER IN FOOD**

***Polytechnic University of Timișoara, Romania***

**Banat's University of Agricultural Sciences and  
Veterinary Medicine "King Mihai I of Romania" -  
Timișoara, Romania**

**May 25- 27, 2014**

**Organised by**



University of Agricultural Sciences and  
Veterinary Medicine "King Mihai I of  
Romania" - Timișoara



*Polytechnic University of Timișoara*



University of Hohenheim





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## **SCHEDULE**

### **Sunday, 25th of May, 2014**

17:00–19:30 Registration of the participants ("*Timișoara*" Hotel)

19:30–19:35 Welcoming words ("*Timișoara*" Hotel)

19:35–21:00 Get-together party ("*Timișoara*" Hotel)

### **Monday, 26th of May, 2014**

9:00–9:20 *Opening of the conference* (Rectors of the *Polytechnic* and *Banat's* universities)

#### ***Session I: Water properties and interactions with biological molecules***

9:20–10:00 Plenary Lecture **PL1**

10:00–10:20 Oral Communication **OC1**

10:20–10:40 Oral Communication **OC2**

10:40–11:30 ***Coffee break, Posters & Exhibition***  
***("Timișoara" Hotel)***

11:30–11:50 Oral Communication **OC3**

11:50–12:10 Oral Communication **OC4**

12:10–12:30 Oral Communication **OC5**

12:30–14:30 ***Lunch ("Belvedere" Restaurant at the***  
***"Timișoara" Hotel)***

14:30–15:10 Plenary Lecture **PL2**

15:10–15:30 Oral Communication **OC6**

15:30–15:50 Oral Communication **OC7**

15:50–16:40 ***Coffee break, Posters & Exhibition***  
***("Timișoara" Hotel)***

**Session II: Water and food stability and safety**

16:40–17:00 Oral Communication **OC8**

17:00–17:20 Oral Communication **OC9**

17:20–17:40 Oral Communication **OC10**

19:00– **Conference Dinner (Recaș Winery,**  
**<http://www.recaswine.ro/index.php?r=ie>)**

**Tuesday, 27th of May, 2014**

**Session III: Water content and water activity of food**

9:00–9:40 Plenary Lecture **PL3**

9:40–10:00 Oral Communication **OC11**

10:00–10:20 Oral Communication **OC12**

10:20–11:30 **Coffee break, Posters & Exhibition,**  
**"The Best Poster" Selection ("Timișoara" Hotel)**

11:30–11:50 Oral Communication **OC13**

11:50–12:10 Oral Communication **OC14**

12:10–12:30 Oral Communication **OC15**

12:30–14:30 **Lunch ("Belvedere" Restaurant at the**  
**"Timișoara" Hotel)**

14:30–14:50 Oral Communication **OC16**

14:50–15:10 Oral Communication **OC17**

15:10–15:30 Oral Communication **OC18**

15:30–16:00 **Concluding remarks & closing of the conference**

## SCIENTIFIC PROGRAMME

### 1st Day – Monday, 26th of May, 2014

- 9:00–9:20 **Opening of the conference**  
**Prof. Corneliu-Mircea DAVIDESCU** (Polytechnic University of Timișoara, Romania)  
**Prof. Paul PÂRȘAN** (Banat's University of Agricultural Sciences and Veterinary Medicine "King Mihai I of Romania" - Timișoara, Romania)
- Session I: Water properties and interactions with biological molecules**
- Chaired by: **Prof. Mohamed MATHLOUTHI**  
(University of Champagne-Ardenne, France)
- 9:20–10:00 **PL1: THE FIVE MECHANISMS OF WATER-SOLID INTERACTIONS: MEASUREMENT AND CASE STUDIES**  
**Lisa J. MAUER**, Matthew ALLAN, Seda ARIOGLU, Belinda CHRISTINA, Na LI, Krystin MARRS
- 10:00–10:20 **OC1: RELATION BETWEEN MOISTURE TRANSFER, WATER WETTABILITY AND STRUCTURE OF GELATIN BASED FILMS TREATED OR NOT BY ELECTRON BEAM**  
**Nasreddine BENBETTAÏEB**, Thomas KARBOWIAK, Salwa BORNAZ, Andrée VOILLEY, Frédéric DEBEAUFORT
- 10:20–10:40 **OC2: HYDRATION AND APPARENT VOLUMINOSITY OF CASEIN MICELLE SUSPENSIONS**  
**Stefan NOEBEL**, Joerg HINRICHS
- 10:40–11:30 **Coffee break, Posters & Exhibition ("Timișoara" Hotel)**
- 11:30–11:50 **OC3: CALCIUM AND ASCORBIC ACID AFFECT CELLULAR STRUCTURE AND WATER MOBILITY IN APPLE TISSUE DURING OSMOTIC DEHYDRATION IN SUCROSE SOLUTIONS**  
**Maria A. MAURO**, **Nicolò DELLAROSA**, Urszula TYLEWICZ, Silvia TAPPIA, Luca LAGHI, Pietro ROCCULI, Marco DALLA ROSA

- 11:50–12:10 **OC4:** THE IMPACT OF AMYLASES ON WATER AND BIOPOLYMER DYNAMICS DURING STORAGE OF STRAIGHT-DOUGH WHEAT BREAD  
**G.M. BOSMANS, B. LAGRAIN, E. FIERENS, J.A. DELCOUR**
- 12:10–12:30 **OC5:** RELATIVE IMPORTANCE OF MOISTURE MIGRATION AND AMYLOPECTIN RETROGRADATION TO POUND CAKE CRUMB FIRING  
**A. LUYTS, E. WILDERJANS, I. VAN HAESENDONCK, K. BRIJS, C.M. COURTIN, J.A. DELCOUR**
- 12:30–14:30 **Lunch ("Belvedere" Restaurant at the "Timișoara" Hotel)**

**Session I: Water properties and interactions with biological molecules**

Chaired by: **Assoc. Prof. Daniel HĂDĂRUGĂ**  
(Polytechnic University of Timișoara, Romania)

- 14:30–15:10 **PL2:** WATER STRUCTURE AND INTERACTIONS IN FOOD, COSMETICS AND PHARMACEUTICALS  
**Mohamed MATHLOUTHI**
- 15:10–15:30 **OC6:** THE EFFECTS OF COMPOSITION AND WATER PLASTICIZATION ON FLUIDNESS PROPERTIES OF DAIRY POWDERS  
**Runjing LI, Mark FENELON, Yrjö H. ROOS, Song MIAO**
- 15:30–15:50 **OC7:** WATER VAPOR SORPTION AND NEW INSIGHTS IN HYDROPHILIC – HYDROPHOBIC INTERACTION  
**Jürgen ADOLPHS**
- 15:50–16:40 **Coffee break, Posters & Exhibition ("Timișoara" Hotel)**



**Session II: Water and food stability and safety**

- 16:40–17:00 **OC8:** A COMPARISON OF ANALYTICAL TECHNIQUES USED TO DETERMINE THE DELIQUESCENCE POINT OF CRYSTALLINE INGREDIENTS  
*Matthew ALLAN, Lisa J. MAUER*
- 17:00–17:20 **OC9:** KINETICS OF SWELLING PROCESS AND RELEASE MECHANISMS OF CORIANDER SATIVUM L ESSENTIAL OIL FROM CHITOSAN/ALGINATE/INULIN MICROCAPSULES IN WATER  
*Cristian DIMA, Livia PATRASCU, Alina CANTARAGIU, Petru ALEXE, Ștefan DIMA*
- 17:20–17:40 **OC10:** INFLUENCE OF THE GRIST / BREWING WATER RATIO ON THE TECHNOLOGICAL POTENTIAL OF SOME SPECIAL RYE MALT MASHES FOR THE BREWING INDUSTRY  
*Mariana-Liliana PĂCALĂ, Lidia FAVIER, Yassine KADMI, Diana STEGĂRUȘ, Otto KETNEY, Ovidiu TIJA*
- 19:30 **Conference Dinner (Recaș Winery, <http://www.recaswine.ro/index.php?r=ie>)**

**2nd Day – Tuesday, 27th of May, 2014**

**Session III: Water content and water activity of food**

Chaired by: **Prof. Lisa MAUER** (Purdue University, USA)

- 9:00–9:40 **PL3:** THE PROBLEM OF WATER CONTENT DETERMINATION IN PRODUCTS CONTAINING LACTOSE  
*Heinz-Dieter ISENGARD, Georg MERKH*
- 9:40–10:00 **OC11:** A NEW METHOD TO ESTABLISH ROBUST SORPTION ISOTHERMS  
*Vincent MEUNIER, M. DUPAS-LANGLET, L. FORNY, S. SAMAIN, M.I. GIARDIELLO*
- 10:00–10:20 **OC12:** EFFECT OF WATER ACTIVITY AND SYSTEM MOBILITY ON PECTINESTERASE ACTIVITY IN SOLUTION  
*Giampiero SACCHETTI, Lilia NERI, Carla D. DI MATTIA, Dino MASTROCOLA, Paola PITTIA*
- 10:20–11:30 **Coffee break, Posters & Exhibition, "The Best Poster" Selection ("Timișoara" Hotel)**

**Session III: Water content and water activity of food**

Chaired by: **Prof. Heinz-Dieter ISENGARD**  
(University of Hohenheim, Germany)

- 11:30–11:50 **OC13:** MICROWAVE RESONANCE MEASUREMENT OF GRAIN  
**Udo SCHLEMM**, Hendrik RICHTER, Rainer HERRMANN
- 11:50–12:10 **OC14:** STUDY OF WATER FEATURES IN COFFEE BEANS BY USING DYNAMIC DEWPOINT AND DIELECTRIC METHODS  
**Pietro ROCCULI**, Eleonora IACCHERI, Chiara CEVOLI, Annachiara BERARDINELLI, Luigi RAGNI, Santina ROMANI
- 12:10–12:30 **OC15:** RAPID TD-NMR (TIME-DOMAIN NUCLEAR MAGNETIC RESONANCE) METHOD FOR THE SIMULTANEOUS DETERMINATION OF FAT AND MOISTURE IN PREDOMINANTLY DRY FOODSTUFFS  
**Steven P. HAILEY**, Devin DARRELL, Colin L. SIMPSON
- 12:30–14:30 **Lunch ("Belvedere" Restaurant at the "Timișoara" Hotel)**
- 14:30–14:50 **OC16:** FLAVONOID AND FLAVONOID-FATTY ACID BIOCONJUGATE / CYCLODEXTRIN COMPLEXES: A KARL FISCHER WATER TITRATION APPROACH  
**Daniel I. HĂDĂRUGĂ**, Nicoleta G. HĂDĂRUGĂ, Heinz-Dieter ISENGARD
- 14:50–15:10 **OC17:** ANALYSIS OF WATER CONCENTRATION OF THERMALLY DEGRADED PALM OIL BY PRE-FROZEN POTATOES AND CHICKEN NUGGETS BY KARL FISCHER TITRATION  
**Dan-Ștefan CLONDA**, Adrian RIVIȘ, Nicoleta G. HĂDĂRUGĂ
- 15:10–15:30 **OC18:** KARL FISCHER WATER TITRATION PARAMETERS OF RYE AND WHEAT FLOUR MIXTURES  
**Corina I. COSTESCU**, Teodor TRĂȘCĂ, Ioan DAVID, Nicoleta G. HĂDĂRUGĂ
- 15:30–16:00 **Concluding remarks & closing of the conference**

**POSTER SESSION:**

**Session I: Water properties and interactions  
with biological molecules**

**P1:** WATER STATUS AND MOBILITY AFFECT THE IN VITRO ANTIOXIDANT ACTIVITY OF MODEL FOOD SYSTEMS CONTAINING BIOACTIVE COMPOUNDS

*Carla D. DI MATTIA, Lilia NERI, Dino MASTROCOLA, **Giampiero SACCHETTI***

**P2:** RELATION BETWEEN MOISTURE SORPTION AND MECHANICAL PROPERTIES OF STARCH-OIL LAMINATE FILMS

*Ewelina BASIAK, Frédéric DEBEAUFORT, Andrzej LENART, **Andrée VOILLEY***

**P3:** POTATO FIBRE INFLUENCE ON WATER STATUS AND STALING OF BREAD

*Eleonora CARINI, Elena CURTI, **Agoura DIANTOM**, Elena VITTADINI*

**P4:** DIFFERENT BEHAVIOR OF WATER IN FRUIT FILLINGS PREPARED WITH THE ADDITION OF INULIN, PECTIN AND GELLAN GUM

*Janna CROPOTOVA, Urszula TYLEWICZ, Nicolò DELLAROSA, **Santina ROMANI** & Marco DALLA ROSA*

**P5:** WATER CONTRIBUTION TO THE STRUCTURATION OF STARCH MATRICES IN THE PRESENCE OF FLAVOUR

***Silawan SOMBOONCHAN**, Samuel LUBBERS, Gaëlle ROUDAUT*

**P6:** PHYSICOCHEMICAL CHARACTERISATION OF CARVACROL- $\beta$ -CYCLODEXTRIN INCLUSION COMPLEXES

*Adriano Antunes de Souza ARAÚJO, Paula dos Passos MENEZES, **Mairim Russo SERAFINI**, Lucindo José QUINTANS-JÚNIOR, Bruno Vasconcelos de SANTANA, Polliana Barbosa Pereira dos SANTOS, Francilene Amaral da SILVA, Gabriel Francisco da SILVA*

**P7:** SILYBIN, RUTIN AND THEIR FATTY ACID BIOCONJUGATE/CYCLODEXTRIN COMPLEXES AS POTENTIAL HEPATOPROTECTIVE AGENTS. A KARL FISHER WATER TITRATION APPROACH

**Daniel I. HĂDĂRUGĂ**, Noemi SANTA, Cătălina CHERA, Lenuța Maria ȘUTA, Gerlinde RUSU, Nicoleta G. HĂDĂRUGĂ, Heinz-Dieter ISENGARD

**P8:** INFLUENCE OF FLAVONOID GLYCOSIDE NARINGIN AND HESPERIDIN FATTY ACID BIOCONJUGATION ON THE WATER CONTENT OF THEIR CYCLODEXTRIN COMPLEXES

**Daniel I. HĂDĂRUGĂ**, Emilia TĂMĂȘOIU, Angelica BOGDAN, Geza N. BANDUR, Nicoleta G. HĂDĂRUGĂ, Heinz-Dieter ISENGARD

**P9:** ADSORPTION BEHAVIOR OF BULGUR

E. AYKIN, **M. ERBAS**, S. ARSLAN, A.N. DURAK

**P10:** SUPERHEATING STEAM COOKING TO ENHANCE FLAVOURS OF POTATOES

Emilie DESCOURS, Jean-Marie DELAITRE, Frédéric DEBEAUFORT, **Andrée VOILLEY**, Anne-Marie SEUVRE

**P11:** EFFECT OF WATER AND GLUTEN ON PHYSICO-CHEMICAL PROPERTIES AND STABILITY OF READY TO EAT SHELF-STABLE PASTA

**Agoura DIANTOM**, Eleonora CARINI, Elena CURTI, Fabrizio CASSOTTA, Alessandro D'ALESSANDRO, Elena VITTADINI

## **Session II: Water and food stability and safety**

**P12:** EVALUATION OF THE EFFECT OF TWO ANTIOXIDANTS FORMULATIONS ON CHEMICAL, PHYSICAL AND MICROBIOLOGICAL PROPERTIES OF A KIWI JAM DURING TWENTY WEEKS OF STORAGE

Célia PIMENTA, **Manuela VAZ VELHO**, Luís PATARATA, Rita PINHEIRO

**P13:** STUDY OF THE EFFECT OF PH ON COLOUR OF FLAVOURED SOLUTIONS OF SPIRULINA SPP. AND CHLORELLA VULGARIS

A. Catarina CORREIA, Pilar MORAIS, M. Helena GOMES, Carla BARBOSA, **Manuela VAZ VELHO**

**P14:** THE ANTIMICROBIAL MECHANISM OF ELECTROLYZED OXIDIZING WATER AND ITS ROLE IN THE FOOD INDUSTRY. AN OVERVIEW

**Ana-Maria CIUCIU**, *Camelia VIZIREANU, Petru ALEXE, Maricica STOICA*

**P15:** PHTHALATES CONTAMINANTS IN BOTTLED WATER

**Luiza GĂINĂ**, *Castelia CRISTEA, Emese GAL, Luminita SILAGHI-DUMITRESCU*

**P16:** CARP MIOFIBRILAR PROTEIN CONCENTRATE DRY BY USING SPRAY DRYER TECHNOLOGY AND ELEMENTAL MAPPING OF MICROSTRUCTURES BY SCANNING ELECTRON MICROSCOPY

**Florice! CERCEL**, *Mariana STROIU, Petru ALEXE*

**P17:** INVESTIGATION OF WATER QUALITY FOR FOOD PROCESSING: MONITORING OF THE NITROSAMINES CONTENT BY ULTRA HIGH PERFORMANCE LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY

*Y. KADMI, L. FAVIER, A.I. SIMION, M.L. PĂCALĂ, C. GRIGORAS, D. WOLBERT*

**P18:** THE INFLUENCE OF THE STORAGE MICROCLIMATE ON THE FRESHNESS OF THE APPLES, DETECTED THE ELECTRONIC NOSE

**Mirela CALU**, *Petru ALEXE*

**P19:** WATER QUALITY USED IN THE TECHNOLOGICAL PROCESS OF OBTAINING BEER

**Ana Maria DODOCIOIU**, *Mirela CALUTU, M. DODOCIOIU, Daniela CIUPEANU, Carmen VLADULESCU*

### **Session III: Water content and water activity of food**

**P20:** COMMON-ION EFFECTS ON DELIQUESCENCE LOWERING OF CRYSTALLINE INGREDIENT BLENDS

*Matthew ALLAN, Lisa J. MAUER*

**P21:** MEASURING WATER VAPOR PERMEATION FOR FOOD PACKAGE MATERIALS USING CRDS

**Byung Il CHOI**, Sang Bong WOO, Jong Chul KIM, Sang-Wook LEE

**P22:** INVESTIGATION OF WATER FEATURES OF BISCUIT DURING STORAGE THROUGH THE USING A RAPID DYNAMIC DEWPOINT METHOD

**Santina ROMANI**, **Pietro ROCCULI**, Silvia TAPPI, Marco DALLA ROSA

**P23:** INFLUENCE OF DIFFERENT DRYING METHODS ON THE PHYSICO-CHEMICAL PROPERTIES OF PUMPKIN

**Liliana SEREMET (CECLU)**, Elisabeta BOTEZ, Oana-Viorela NISTOR, Doina Georgeta ANDRONOIU, Gabriel-Dănuț MOCANU

**P24:** THE INFLUENCE OF PHOSPHOLIPASES ON THE WATER CONTENT OF VARIOUS BREAD TYPES

**Laura CORPAȘ**, Paul PÎRȘAN, Ioan DAVID, Nicoleta G. HĂDĂRUGĂ, Heinz-Dieter ISENGARD

**P25:** A STUDY REGARDING PRESERVATION PERIOD OF A DIETETIC MEAT PRODUCT

**Cristian TUDOSE**, Livia PATRASCU, Petru ALEXE

**P26:** WATER CONTENT OF TRITERPENOID SAPONINS AND THEIR FATTY ACID BIOCONJUGATE/CYCLODEXTRIN COMPLEXES

**Corina VASILESCU**, Nicoleta G. HĂDĂRUGĂ, Gerlinde RUSU, Daniel I. HĂDĂRUGĂ



## **ORAL PRESENTATIONS**







**PL 1**

**THE FIVE MECHANISMS OF WATER-SOLID INTERACTIONS:  
MEASUREMENT AND CASE STUDIES**

**Lisa J. MAUER**, Matthew ALLAN, Seda ARIOGLU,  
Belinda CHRISTINA, Na LI, Krystin MARRS

*Department of Food Science, Purdue University, West Lafayette, USA*

There are five major mechanisms of water-solid interaction: adsorption onto the surface of the solid particle, capillary condensation, crystal hydrate formation, deliquescence, and vapor absorption into the bulk of amorphous solids. Recognition of the mechanisms by which water can interact differently with crystalline and amorphous solids, and the different amounts of water brought in by these mechanisms, has overcome some formulation challenges; however, as foods increase in complexity beyond single crystalline or amorphous ingredients, the moisture transfer between ingredients and resulting physical and chemical changes become important. A summary of moisture sorption results for crystalline, amorphous, and blended crystalline-amorphous food ingredient systems across multiple temperatures obtained by a variety of measurement techniques (including static isopiestic methods in relative humidity [RH] controlled desiccators, and a variety of gravimetric moisture sorption instruments: Project Messtechnik SPS-x, Decagon Devices VSA and AquaSorp, VTI) will be presented. The relationships between environmental RH and the moisture sorption rate as well as heat of solution, solubility, and deliquescence RH, for deliquescent organic solids and blends thereof will be described. RH-temperature phase diagrams of deliquescent crystalline ingredients that are able to form hydrate structures will be discussed. Finally, the synergistic moisture uptake and resulting effects on the glass transition temperature and dissolution in co-formulated crystalline and amorphous ingredient blends will be revisited.

**PL 2**

**WATER STRUCTURE AND INTERACTIONS IN FOOD,  
COSMETICS AND PHARMACEUTICALS**

**Mohamed MATHLOUTHI**

*Université de Reims Champagne Ardenne,  
Association Andrew VanHook, Reims, France*

Liquid water structure is known to be that of an associated liquid involving different species of associates by H-bonding. The presence of small hydrophilic solutes such as glycerol or carbohydrates affects the hydrogen bonding in solution and the functional properties of the food, the cosmetic or pharmaceutical product.

After recalling of the properties of hydrogen bonds, of hydrophilic and hydrophobic hydration, the effect on water structure of sweet (sucrose) and bitter (caffeine) molecules is reported and the effect of sucrose concentration on masking of the bitter taste of caffeine analyzed. Likewise the effect of some inhibitors the unpleasant taste of nicotine is derived from surface tension studies of mixtures of nicotine-inhibitor solutions in dilute aqueous solutions.

In cosmetics, the skin hydration is known to be an important issue. Hydrating agents used as moisturizers are reviewed. Of particular relevance is glycerol which is frequently used as humectant. The balance between hydrophilic and hydrophobic interactions of the glycerol molecule helps in understanding the efficiency of this small humectant as skin moisturizer. Other types of humectants are presented with reference to their hydrophilic or lipophilic interaction with water.

Water vapor also interferes in the storage stability of food and pharmaceuticals. This aspect of water effect on food and pharmaceuticals is described by water vapor sorption isotherms and the value of water activity of the investigated product. The caking of food powders, especially granulated sugar is reported as an example. The amorphous state of food powders might be at the origin of caking. However, there is a need of use of amorphous particles together with polymers and the right amount of hydration water in order to successfully perform the tableting of pharmaceuticals.

PL 3

**THE PROBLEM OF WATER CONTENT DETERMINATION IN PRODUCTS CONTAINING LACTOSE**

**Heinz-Dieter ISENGARD**, Georg MERKH

*University of Hohenheim, Institute of Food Science and Biotechnology,  
Stuttgart, Germany*

The water content of pure lactose (any isomer) can be determined both by drying as by Karl Fischer titration. The water of crystallisation of  $\alpha$ -lactose as lactose monohydrate is released only at relatively high temperatures. This fact may lead to serious problems when the water content of products containing lactose is to be determined. In some cases, the determination of water content by drying becomes even impossible. With the Karl Fischer titration combined with gas extraction it is possible to make such a situation visible. The sample water (and possibly other volatile substances) is driven by a dry air stream from the vial containing the sample into the Karl Fischer titration cell, where water is determined selectively. The vial can be heated using constant temperature or a temperature ramp. The detection rate of water and the total water detected are monitored. This technique allows in critical situations to show that the "lactose water" is only detected at temperatures at which the degradation of substances in the sample has already begun. This overlapping proves that a correct water determination by drying is impossible for such a product. No temperature exists where on the one hand the water of lactose is completely released and on the other hand the matrix does not release water produced by degradation. Baby food is presented as practical example for such a situation. As the Karl Fischer titration does not use such high temperatures, it is the method of choice in these cases. It is remarkable that, neglecting this fact and against scientific evidence, an official method to determine moisture of milk powder by drying has been established and still exists.

OC 1

**RELATION BETWEEN MOISTURE TRANSFER, WATER WETTABILITY AND STRUCTURE OF GELATIN BASED FILMS TREATED OR NOT BY ELECTRON BEAM**

**Nasreddine BENBETTAÏEB**<sup>1,2</sup>, Thomas KARBOWIAK<sup>2</sup>,  
Salwa BORNAZ<sup>3</sup>, Andrée VOILLEY<sup>2</sup>, Frédéric DEBEAUFORT<sup>2,4</sup>

<sup>1</sup> National Center for Nuclear Sciences and Technologies, Ariana, Tunisia

<sup>2</sup> AgrosupDijon, Dijon, France

<sup>3</sup> Higher Institute of Food Industries of Tunis, El Khadra, Tunisia

<sup>4</sup> University of Burgundy, Dijon, France

Gelatin has relatively low cost and excellent functional and filmogenic properties<sup>1</sup>. It has also been extensively studied in its use as an outer covering to protect food against light and oxygen as edible films and coatings. However, this polymer is highly sensitive to moisture and exhibits poor water vapour barrier properties<sup>2</sup>. The cross-linking of polymers by means of chemical, enzymatic, or physical treatments was reported to improve the barrier as well as the mechanical properties by the formation of a denser network through chemical binding between polymers. A promising alternative consists in crosslinking polymers by using high-energy ionizing radiation, such as electron beam, gamma, or X-ray. The aim of this work is to display the impact of irradiation on the structure of gelatin films, studied by DSC, XRD and TGA, and on the water wettability as well as on water vapour permeability.

Irradiation induced a slight but significant increase of WVP of about 12% as well as the contact angle of water decreased, indicating a greater sensitivity to water. The effect of the irradiation dose seems to be not important, as the change occurred even for the lower dose (20 kGy) and no differences observed between 20 and 60 kGy. Data obtained by DSC displayed an increase of the glass transition value that could be related to an anti-plasticization action of the irradiation, which seems contradictory to moisture transfer and wetting of gelatin films. DRX and TGA results display more complex effect of irradiation on gelatin structure.

**References:**

- <sup>1</sup>N.Cao;Y.Fu;J.He, Preparation and physical properties of soy protein isolate and gelatin composite films. *Food Hydrocolloids*.**2007**, 21, 1153–1162.
- <sup>2</sup>Ben Bettaieb N., Kurek M., Bornaz S., Debeaufort F. Barrier, structural and mechanical properties of bovine gelatin/chitosan blend films related to biopolymer interactions, *J. Sci. Food Agric.*, **2014**, DOI: 10.1002/jsfa.6570

OC 2

**HYDRATION AND APPARENT VOLUMINOSITY OF CASEIN MICELLE SUSPENSIONS**

**Stefan NOEBEL<sup>1</sup>**, Joerg HINRICHS<sup>1</sup>

<sup>1</sup> *Institute of Food Science and Biotechnology, University of Hohenheim, Stuttgart, Germany*

Caseins are the major proteins in bovine milk and are present as casein micelles with an average diameter of about 50–300 nm. They are colloidally dispersed in the aqueous phase of the milk. All assembly models of casein micelles have in common that the casein monomers arrange as spherical particles and bind water inside and around the substructure. The large amount of water being bound is due to the shell of the  $\kappa$ -caseins which form a polyelectrolyte brush. An important property of casein micelles, independent of their detailed structure, is their ratio of hydration. The amount of bound water depends upon temperature, pH and ionic strength.

The term voluminosity in general is used for the spherical volume, including the excess water bound in the rough particle surface that influences the physico-chemical properties of this particle (hydrodynamic volume). Beyond that, there exists a hydrodynamically influenced volume, which acts like one particle in rheological measurements.

A new method to determine the effect of temperature on the apparent voluminosity of casein micelles by means of rheometry was proposed and applied. The presented method is independent of the knowledge about the way that water binds and the concentration effects, as long as the viscosity-concentration and voluminosity-temperature relationship follow continuous functions. The observed decrease of the apparent voluminosity with increasing temperature is consistent with literature values. Furthermore, the apparent voluminosity asymptotically reached a plateau which is supposed to be a compensating effect of protein swelling, hydrophobicity, and intra-micellar migration.

OC 3

**CALCIUM AND ASCORBIC ACID AFFECT CELLULAR STRUCTURE AND WATER MOBILITY IN APPLE TISSUE DURING OSMOTIC DEHYDRATION IN SUCROSE SOLUTIONS**

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Osmotic dehydration (OD) of plant foods is largely controlled by the cellular membranes, which present different permeability. When the cellular structure is destroyed, the tissue loses its selectivity and water mobility and distribution are modified. Actually the knowledge about tissue structure and mass transport is fundamental for OD control. The aim of this work was to investigate the effect of addition of calcium lactate (CaLac) and ascorbic acid (AA) to sucrose (Suc) osmotic solutions on cell viability and structure of apple tissue, and to the consequent water distribution and mobility modification on the different cellular compartments. Fluorescence Microscopy (FM), Light Microscopy (LM) and Time Domain Nuclear Magnetic Resonance (TD-NMR) were used. Cell viability was evaluated by fluorescence intensity, using fluorescence in diacetate, and vacuole integrity by neutral red accumulation in preserved vacuoles. Treated tissues in AA-CaLac-Suc solution did not show vitality anymore. Very low fluorescence intensity was detected for AA-Suc treatment, being slightly higher for CaLac-Suc solution, while for Suc solution, intensity was comparable to that observed in fresh tissue. In this sample numerous preserved vacuoles and red-stained cells were visualized, in agreement with FM results. The low influence of Suc treatment on cellular compartmentation and functionality slightly influenced water distribution and mobility in the apple tissue, as highlighted by TD-NMR, evidencing a higher vacuole volumes and lower cytoplasm-extracellular spaces in Suc treated tissue, when compared to the other treatments. AA presence reduced process efficiency (e.g. the ratio between water loss and solute gain) and enhanced Ca impregnation, which was associated to degradation of membranes and, thus, to more spaces viable to solute diffusion.

OC 4

**THE IMPACT OF AMYLASES ON WATER AND BIOPOLYMER DYNAMICS DURING STORAGE OF STRAIGHT-DOUGH WHEAT BREAD**

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Bread is a popular staple food but has a limited shelf life. Fresh bread at ambient temperature contains a semi-crystalline amylose network and a thermoset gluten network. During storage, its crumb firms due to a combination of different events and to a degree determined by the strength of the different networks in the system. Amylopectin recrystallizes in a process referred to as retrogradation. This results in a continuous, rigid, semi-crystalline network which is strongly related to the increase in crumb firmness during the first days of storage. Besides structural changes in the starch fraction, water is also redistributed. It not only diffuses from crumb to crust, but also between biopolymer types. The combination of all of these phenomena stiffens the gluten network when its water content drops below a typical value, leading to a further increase in crumb firmness after a couple of days of storage.

However, the impact of amylopectin retrogradation on the extent of water redistribution between gluten and starch is still under debate. The objective of this study was to investigate the importance of the starch mesoscale network organization for water distribution and, as a result, for crumb texture properties. To that end, three  $\alpha$ -amylases that differently impact the starch (re)crystallization behavior and the resulting network organization were added to the bread recipe. The formation of starch crystals and changes in crumb firmness were monitored. Proton nuclear magnetic resonance was used to analyze proton mobility of the bread constituents. The exo-working amylases to a variable degree slowed down amylopectin retrogradation, resulting in a less prominent starch network that included less water. Dehydration of the gluten network was therefore presumably less pronounced, contributing to a smaller increase in crumb firmness and a smaller decrease in crumb resilience. Internal starch degradation by an endo-working amylase resulted in sticky bread crumb with very low resilience.



OC 5

**RELATIVE IMPORTANCE OF MOISTURE MIGRATION AND  
AMYLOPECTIN RETROGRADATION TO POUND CAKE CRUMB  
FIRMING**

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Moisture migration largely impacts cake crumb firmness during storage at ambient temperature. To study the importance of phenomena other than crumb to crust moisture migration, crustless cakes were baked in an electrical resistance oven (ERO). Proton nuclear magnetic resonance (<sup>1</sup>H NMR) was used to study the storage-induced changes in water distribution in the crumb of cakes baked either conventionally or in the ERO. Crumb firming of cakes baked either conventionally or in an ERO was evaluated. In cakes baked conventionally, most of the increase in crumb firmness during storage was caused by moisture migration from crumb to crust. <sup>1</sup>H NMR measurements on ERO and conventional cake crumb showed that the population containing protons of crystalline starch became more abundant during cake storage. This can be attributed to limited amylopectin retrogradation, as was also confirmed with differential scanning calorimetry (DSC). The limited increase in amylopectin retrogradation during cake storage cannot solely account for the significant firming of ERO cakes. Hence, other phenomena are also involved in cake crumb firming.

**OC 6**

**THE EFFECTS OF COMPOSITION AND WATER  
PLASTICIZATION ON FLUIDNESS PROPERTIES OF DAIRY  
POWDERS**

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Carbohydrate-protein systems are the most important models of dairy solids and food ingredients. The effect of carbohydrate-protein composition on water sorption and fluidness properties including glass forming characteristics and temperature-dependent structural relaxation properties were studied. Lactose, lactose/MPI (milk protein isolate) (4:1, 3:2, 1:1, 2:3, 1:4), and MPI were spray-dried. Brunauer-Emmett-Teller (BET) model was fitted to water sorption data and sorption isotherms were established. Sorbed water increased with storage relative humidity (RH) up to 23.1% RH as well as increased MPI content. Above 44.1% RH, the amount of sorbed water decreased as the content of MPI increased. Water plasticised the dairy powders and lowered glass transition temperature ( $T_g$ ).  $T_g$  of dairy powders has no significant change as the content of MPI increased. The relaxation times were affected by carbohydrate-protein composition and water content. Storage modulus,  $E'$ , of dairy powders had no obvious difference below  $T_g$  when MPI content was increased. But increasing MPI contents decreased the stiffness of dairy powders which was shown by the changes in loss modulus,  $E''$ , above  $T_g$ . Temperature and humidity had a similar effect on the free volume and viscoelastic of dairy powders. Knowledge and understanding of carbohydrate-protein interactions, glass transition, and mechanical relaxations are useful for the control of physical properties of dairy powders and are necessary for the design of complex food and nutrient delivery systems.

OC 7

**WATER VAPOR SORPTION AND NEW INSIGHTS IN  
HYDROPHILIC – HYDROPHOBIC INTERACTION**

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Water vapor interaction on solid surfaces depends strongly on the hydrophilic or hydrophobic materials structure. For example life time, processing and storage properties of food are affected. Recent fundamental investigations including water vapor sorption, calorimetry and reversible adsorption (inverse gas chromatography) on various porous and non-porous model substances either pristine or silanised will be presented. The isotherms can differ quite unexpectedly. For example hydrophobic materials show a higher water uptake per area compared to their pristine hydrophilic counterparts. For hydrophobic materials on the other hand, capillary condensation does not take place as expected, but a water loss at higher relative humidity is again quite surprising. We will give some answers based on the ESW (Excess Surface Work), a modelless method, and analyze the thermodynamics in particular the dispersive and structural forces.

These results are then applied on water vapor sorption isotherms of various foods, like coffee, grains and soup powders in order to characterize their hydrophilic / hydrophobic behavior.

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OC 8

**A COMPARISON OF ANALYTICAL TECHNIQUES USED TO DETERMINE THE DELIQUESCENT POINT OF CRYSTALLINE INGREDIENTS**

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Many common food ingredients (sucrose, NaCl, ascorbic acid, etc.) are deliquescent crystalline solids that undergo dissolution once a critical relative humidity, known as the deliquescence point ( $RH_0$ ), is exceeded. Blends of deliquescent ingredients will have a lower deliquescence point ( $RH_{0mix}$ ) than the single ingredients. A variety of methods have been used to measure deliquescence; however, no extensive comparison of methods has been published to address the effects of experimental variables or the advantages and disadvantages of different techniques for determining the  $RH_0$  and  $RH_{0mix}$ . The objective of this study was to provide a comprehensive comparison of methods used to measure deliquescence points of single ingredients and blends (sucrose, fructose, NaCl, KCl). The following methods were compared: water activity of saturated solutions prepared using different water:solid ratios and different ingredient ratios in blends, dynamic vapor sorption isotherms (DVS) with different RH steps and equilibrium criteria, and dynamic dewpoint sorption (DDI) profiles. Significant differences ( $p < 0.05$ ) in the measured  $RH_0$  and  $RH_{0mix}$  were found between different methods and experimental factors. For the  $a_w$  method, sample preparation recommendations are to use 1-3 grams of sample, add 25-100  $\mu$ L of water per gram of sample, and to wait at least 24 hours before measurement. When preparing ingredient blends for  $a_w$  measurement, blending at the eutonic composition allowed for a greater range of water:solid ratios for a consistent prediction of the  $RH_{0mix}$ . In the DVS methods, the RH step proved to be the most important factor when determining the  $RH_0$ , and the following criteria are recommended: 1% RH steps, 0.01% equilibrium criteria, and 3 hour step time. When investigating the  $RH_{0mix}$  of ingredient blends, the  $a_w$  method gave more consistent results than the DVS and DDI profiles, likely due to the variability of contact points between

different ingredients in the crystal blends. Advantages and disadvantages of the different methods were summarized and could serve as a guide for selecting reliable  $RH_0$  and  $RH_{0mix}$  measurements.

OC 9

**KINETICS OF SWELLING PROCESS AND RELEASE MECHANISMS OF CORIANDER SATIVUM L ESSENTIAL OIL FROM CHITOSAN/ALGINATE/INULIN MICROCAPSULES IN WATER**

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The coriander essential oil is used in the food industry both as flavour compound and preservation agent due to its antioxidant and antimicrobial properties. Depending on their physico-chemical sensitivities, essential oils are encapsulated in different matrices. An important characteristic of microcapsules is the release rate of bioactive components under different conditions.

This paper presents the study of the coriander essential oil release mechanism in water. The coriander essential oil was obtained from seeds through supercritical CO<sub>2</sub> extraction. The encapsulation of the coriander essential oil was carried out through the spray drying method (inlet temperature of 130°C and feed rate of 0.5 L h<sup>-1</sup>) using as encapsulating material chitosan, alginate and inulin in different ratios (24% dried matter). The O/W emulsions were prepared through ultrasonation of biopolymers and coriander essential oil blends in mass ratio, dried matter:oil, of 3:1, in the presence of lecithin as emulsifier. The swelling degree of the microcapsules and the release rate of the coriander essential oil were correlated with the viscosity of the encapsulating material, the water activity, pH and temperature. The power law  $M_t/M_\infty = kt^n$  was used as mathematic model and it was demonstrated that the release of the coriander essential oil from chitosan/alginate/inulin microcapsules follows a first order kinetics and corresponds to a non-Fickian transport phenomenon which depends simultaneously on the swelling of the microcapsule and on the oil diffusion.

OC 10

**INFLUENCE OF THE GRIST / BREWING WATER RATIO ON THE TECHNOLOGICAL POTENTIAL OF SOME SPECIAL RYE MALT MASHES FOR THE BREWING INDUSTRY**

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For the brewing industry, water is the raw material that significantly contributes to the main physical-chemical characteristics of wort and, consequently, of the beer, but especially determines the variant of management of the technological process of brewing. There is very little information in the specialty literature regarding the use of rye malt for special brewing. The main objective of this laboratory scale study was to determine the potential of malt rye (30% and 40%; 6 EBC colour) in combination with *Pilsner* barley malt (70% and 60%), both malts from *Weyermann* Specialty Malting, Bamberg, Germany, for mashing and producing wort by one imposed mash diagram; in the conditions of efficient use of water for depletion of malt spent grains. Hardness of water was less than 5 °dH. We analyzed the influence of the initial ratio grist/brewing water (w/w) (R<sub>1</sub>-1:4.0; R<sub>2</sub>-1:4.5; R<sub>3</sub>-1:5.0; R<sub>4</sub>-1:5.5; R<sub>5</sub>-1:6.0; R<sub>6</sub>-1:6.5) on the technological potential (extract, lautering speed, pH, colour and viscosity of wort, free amino nitrogen content, final apparent attenuation of wort, extract yield, soluble extract in spent grains) of special rye malt mashes for brewing industry. Malt grinding was done with a laboratory disc mill, type *DLFU*, with the disc gap set to 0.5 mm. All analytical determinations were performed at least in triplicate in accordance with *Analytica-EBC* and *MEBAK* Methods or other methods in accordance with these (i.e. monosaccharides and oligosaccharide were determined by gas-chromatography GC-FID). Experimental data obtained were expressed as the mean ± SD and subjected to analysis of variance *ANOVA* and *Duncan's* test at  $\alpha < 0.05$ . Results of this laboratory scale study showed that rye malt

has a considerable potential as adjunct in multi-grain ale and lager beer brewing, even high gravity brewing.



OC 11

**A NEW METHOD TO ESTABLISH ROBUST SORPTION ISOTHERMS**

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Water sorption isotherm is of fundamental importance in food industry to predict the stability of dehydrated amorphous products and to establish moisture content norms. An isotherm is commonly based on large  $a_w$  scale using data collected from dynamic sorption devices or desiccators. Both of these methods have some drawbacks. The most important one is related to lack of knowledge in terms of time required to achieve  $a_w$  equilibrium. In case of dynamic sorption devices, operators have to set arbitrary equilibrium criteria (weight variation for a given time). For desiccators, long equilibration times are generally used, assuming that after few weeks, equilibration is achieved. To overcome this equilibration issue, especially in low  $a_w$  region, a new method was developed. First, samples are deliberately brought out of equilibrium by drying or humidifying them in desiccators for rather short time (few days). Then, these samples are placed in  $a_w$  meter (e.g. Decagon or Novasina) where adapted thermal treatment is directly applied in the measuring chamber. Thermal treatment allows to strongly accelerate water transfer within the samples and to achieve complete  $a_w$  equilibration. The objective of this study is to better understand and optimize such thermal treatment conditions (temperature and time), which deeply depends on water diffusion rate. Consequently, the influence of glass transition temperature, powder texture and molecular composition is discussed. Other equilibration parameters are also taken into account such as sample thickness and ventilation effect used in desiccators. Along the presentation, different dehydrated food model systems are considered: from model maltodextrin powders to whole milk powders or green coffee beans. Thanks to the optimization of this new robust method, it becomes possible to obtain robust sorption isotherms in low  $a_w$  range for most dehydrated amorphous products in less than two weeks of lab work.

OC 12

**EFFECT OF WATER ACTIVITY AND SYSTEM MOBILITY ON  
PECTINESTERASE ACTIVITY IN SOLUTION**

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Enzymatic activity in aqueous solutions is influenced by environmental factors such as system mobility, in terms of viscosity and molecular weight of solute, and by the water macroscopic translational mobility, as described by water activity.

The aim of this study was to investigate the effect of viscosity, water activity ( $a_w$ ) and molecular weight of solutes on the activity of pectinesterase (PE) in buffered solutions (pH 7.5) using two different ligands (glucose and sodium chloride) to modulate  $a_w$  and a thickener (maltodextrin, DE 8.7) to modulate viscosity.

Results showed that  $a_w$  seems to play a major role in inhibiting PE activity contrarily to what was observed for other enzymes (horseradish peroxidase and lactoperoxidase), whose activity was mainly reduced by a viscosity increase and only slightly affected by a water activity decrease.

The increase of viscosity induced by maltodextrin addition generally depleted the enzymatic activity except for concentrated systems at high  $a_w$  value, where a viscosity increase determined an increase of enzymatic activity, which could be ascribed to a 'crowding' effect.

Viscosity and water activity being equal, glucose determined a higher PE inhibition than sodium chloride; this could be explained by the higher molecular weight of the former solute, which reduced the system mobility by increasing the glass transition temperature of the system.

Even though PE activity was influenced by the same physical factors that affect the activity of other enzymes, the relative importance of each factor in the inhibition of enzymatic activity could largely depend on the type of enzyme under investigation.

**OC 13**

**MICROWAVE RESONANCE MEASUREMENT OF GRAIN**

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By use of the microwave resonance method, several characteristics of grain can be measured. The moisture of moving grain can be measured by use of a planar microwave sensor, independent of grain size and density. This application is important for controlling grain dryers. The moisture measurement of grain is limited because of the 'maximum effect of the microwave measurement'. This is an effect related to the permittivity of the material, that limits the range, in which the moisture can be measured. This range can be increased by variation of the measuring frequency.

Furthermore a fast and precise measurement of the mass and moisture of single grain is possible by use of the 3D-microwave resonance technique. A selection of single grain can be carried out this way.

OC 14

**STUDY OF WATER FEATURES IN COFFEE BEANS BY USING  
DYNAMIC DEWPOINT AND DIELECTRIC METHODS**

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The mobility and availability of water in coffee depend on the extent of interactions between the aqueous phase and the biopolymeric matrix and it is responsible of anti-plasticization effects. The increase in the understanding of these aspects in food products is very useful, and it can be approached in different ways.

In this direction, the objectives of this study were:

- i) to compare green and roasted coffee adsorption isotherms obtained with both the rapid dynamic dewpoint method and the traditional saturated salt slurry one;
- ii) to assess a rapid and non-destructive methods based on waveguide spectroscopy in order to evaluate moisture and water activity of green and roasted coffee, by analyzing the obtained spectral data in the 2-3, 5-6 GHz and 17-18 GHz frequency ranges.

The comparison between rapid and traditional methods for isotherm determination showed that above the critical  $A_w$  green coffee exhibited a different behaviour and its hydration becomes strictly time dependent, because of its heterogeneous structure. This phenomenon was not detectable for roasted coffee that showed very similar results with the two different applied methods. At low frequency, the purposed dielectric technique showed high potentiality for the non-destructive estimation of water content of green and roasted coffee. At higher frequency, the obtained signal seems to be more related to water/solid matrix interactions and water activity.

OC 15

**RAPID TD-NMR (TIME-DOMAIN NUCLEAR MAGNETIC  
RESONANCE) METHOD FOR THE SIMULTANEOUS  
DETERMINATION OF FAT AND MOISTURE IN  
PREDOMINANTLY DRY FOODSTUFFS**

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While the use of TD-NMR for the measurement of moisture and fat in predominantly dry foodstuffs is well-established, its widespread acceptance for process control and product testing in the food industry has been hindered both by the typically long total analysis times required for sample temperature conditioning as well as the often limited robustness of moisture calibrations. Owing to the requirement that samples be conditioned to defined temperatures, NMR methods have traditionally involved a time consuming pre-conditioning step often exceeding 20 minutes. We describe an approach (patent pending) whereby samples can be rapidly conditioned by conductive heating for short exposure periods to temperatures above that of the target sample temperature. Determination of exposure periods and temperatures is made using an iterative routine that compares the NMR signal obtained following a given rapid equilibration period to that following a period of conventional temperature equilibration. This approach can effectively reduce the required temperature equilibration period from more than 20 minutes to less than 1-2 minutes with no loss in measurement repeatability or accuracy. In addition, while straightforward approaches for moisture determination in predominantly dry foodstuffs have been previously demonstrated, we have found that these approaches are limited by the need to create separate calibrations for sample types which have varying fat/oil content. This is due to the partial NMR relaxation of fat/oil, which effectively interferes with the moisture determination and degrades the linearity of calibration curves containing samples with variable fat/oil content. We describe a mathematical approach (patent pending) whereby the NMR relaxation of fat/oil over variable concentration ranges can be accounted for, thus enabling the use of fewer calibration curves as

well as improving accuracy. Data will be presented to illustrate expected measurement performance using the described approaches for the analysis of several types of dry food products.

OC 16

**FLAVONOID AND FLAVONOID-FATTY ACID BIOCONJUGATE /  
CYCLODEXTRIN COMPLEXES: A KARL FISCHER WATER  
TITRATION APPROACH**

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Flavonoids are very known compounds from plants having a wide range of biological and pharmacological properties, including antioxidant activity and cancer prevention. Protection and controlled release of these valuable compounds can be performed by molecular encapsulation in cyclodextrins. On the other hand, flavonoids (especially flavonoid glycosides) are difficult to encapsulate in cyclodextrins due to their less hydrophobic characteristics [1]. Enhancing the hydrophobicity of flavonoside glycosides can be achieved by fatty acid bioconjugation using enzymatic synthesis. These bioconjugates can be better encapsulated in cyclodextrins and conduct to new formulations with potential applications to functional foods and/or pharmaceutical drugs. The molecular encapsulation process can be evaluated by means of "real" water content of complexes, which can be better performed by using the Karl Fischer titration (KFT) approach.

Enzymatic synthesis of flavonoid-fatty acid bioconjugates was performed in acetone by using Novozyme 435. The cyclodextrin complexation was achieved by crystallisation from ethanol-water solution. The flavonoid and their fatty acid bioconjugate/cyclodextrin complexes were analyzed by DSC and TG, as well as by KFT in order to evaluate the water content and further the quality of the encapsulation process. The importance of the determination of the "correct" water content of food and pharmaceutical grade flavonoid and fatty acid bioconjugate/cyclodextrin complexes is emphasized in this presentation.

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OC 17

**ANALYSIS OF WATER CONCENTRATION OF THERMALLY DEGRADED PALM OIL BY PRE-FROZEN POTATOES AND CHICKEN NUGGETS BY KARL FISCHER TITRATION**

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Potatoes and chicken nuggets are the most consumed foodstuffs in Europe, especially in the fast-food restaurants. The quality of potatoes and nuggets is related to the vegetable oil used for frying, in this case palm oil, which has good stability against oxidation; the oxidation process conducts to more polar compounds and a lower overall hydrophobicity of used vegetable oils. As a result, the water concentration in these oils can increase and have further interactions at frying conditions.

The aim of this paper was to evaluate the modification of water concentration in palm oil which was thermally degraded by frying vegetable products namely pre-frozen potatoes and animal products chicken nuggets. The degradations were conducted in a deep fryer in which a determined quantity of palm oil and then subsequently the products of vegetable and animal origin was inserted. The frying temperature was set at 160 °C for 5 minutes, and after that oil samples were taken after each frying cycle for further evaluation. Karl Fischer titration (KFT) was used for monitoring the water content of the thermally degraded palm oil samples. The water content was relatively low, with values not exceed 0.1%. However, this parameter slowly increase with the frying cycles and well correlates with the other physico-chemical parameters of the palm oil samples (such as relative density, acid, peroxide and iodine indices, as well as total polar fraction determined by classical methods).

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**OC 18**

**KARL FISCHER WATER TITRATION PARAMETERS OF RYE AND WHEAT FLOUR MIXTURES**

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Rye flour is one of the most used material for obtaining "special" bread products in Romania. Unfortunately, this rye flour is often combined with other flour types (such as wheat flour) for economic reasons and thus lowering the overall quality of the food products.

In this study the influence of addition of wheat flour to rye flour for obtaining the raw material for special bread products was studied by means of the determination of water content and types of water binding using the Karl Fischer water titration method. Thus, rye and wheat flour mixtures at ratios ranged from 0% to 100% were analyzed. The KFT parameters (water content, water reaction/diffusion rates) were used in order to discriminate/classify the rye/wheat flour samples.

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## **POSTER PRESENTATIONS**





**WATER STATUS AND MOBILITY AFFECT THE *IN VITRO*  
ANTIOXIDANT ACTIVITY OF MODEL FOOD SYSTEMS  
CONTAINING BIOACTIVE COMPOUNDS**

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Solvent composition, system mobility and viscosity are known to play a central role in affecting the rate of both chemical and biochemical reactions but the effect of these variables on the activity of antioxidants is a neglected area in food research.

The aim of this work was to study the effects of water activity ( $a_w$ ) and mobility of model food systems containing ascorbic acid and catechin on their *in vitro* antioxidant activity.

Physical properties such as  $a_w$ , viscosity, and the molecular weight of co-solutes in the model systems containing bioactive compounds were modulated through ingredients largely used in the food industry: sodium chloride (NaCl), sorbitol (S), glucose (G), maltose (M) and trehalose (T) to deplete water activity whilst the viscosity was modified by using maltodextrin with 8.7 DE (MD). The *in vitro* antioxidant activity was measured by means of the 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) radical decolorization assay (ABTS).

The antiradical activity was significantly affected by water activity and molecular mobility, as described by bulk viscosity, and molecular weight of co-solutes, as described by  $T'g$ . The effect was strictly dependent on both the bioactive compounds tested and on the co-solutes used to modify the physical properties of the model system.

The results obtained in this study suggest an effect of water status and mobility of foods on their *in vitro* antioxidant activity and this effect is often overlooked when measuring the antioxidant activity of foods with high soluble solutes content like honey, jams, and syrups.

## RELATION BETWEEN MOISTURE SORPTION AND MECHANICAL PROPERTIES OF STARCH-OIL LAMINATE FILMS

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**Introduction:** Water content in edible films plays a very important role. Applications of plastic material replacements change according to the H<sub>2</sub>O quantity. Also depends on water content and activity changes kind of using materials.

The objective of this study was to investigate sorption properties of laminate films based on wheat starch and rapeseed oil in two starch concentration: 3 (3L) and 5% (5L), including: sorption kinetics, water vapour permeability, moisture sorption isotherms, surface and mechanical properties.

**Results:** The surface free energy was 63.70 (3L) and 56.06 (5L) (mN/m), critical surface tension 40.60 (mN/m) for both, contact angle was 67 and 77° for 3L and 5L films, respectively. Non-polar interactions were much stronger than polar interactions. The non-polar energy was about 3 and 4 times higher than polar energy. Water vapour permeability rises significantly with the humidity differential. The shape of the water sorption isotherm is not strongly affected by the oil coating. SEM photographs display single oil drops in starch matrix on both surface and cross section. Elongation of films increases with starch content whereas tensile strength is reduced, but young modulus remains constant.

Starch-oil laminates films have good wetting properties. Oil droplets are suspended/dispersed in the starch matrix. On the whole surface of films there is no hydrophobic lipid barrier. Films are rigid and have weak mechanical properties.

## POTATO FIBRE INFLUENCE ON WATER STATUS AND STALING OF BREAD

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Potato fibre was added to a white bread formulation to investigate water status and physico-chemical properties during staling. To white bread (STD, flour 100, water 59, sugar 4, seeds oil 3, yeast 3, salt 2) 0.04% of potato fibre (flour basis) was added to produce P-STD (water 59) and P-W (water 64) samples. Samples were characterized fresh and during 7 days of storage for texture (hardness 40% compression), water activity, moisture content (oven drying at 105°C to constant weight), frozen water content and amylopectin retrogradation (DSC analysis, from -80 °C to 130 °C, 5 °C/min), and proton molecular mobility by low resolution <sup>1</sup>H NMR (20 MHz, Free Induction Decay and T<sub>2</sub>). Hardness was comparable among fresh samples, while at day 7 P-W was the softest sample (2.6 N), followed by P-STD (3.7 N) and STD (4.5 N). Moisture content and water activity in fresh sample crumb were ~40% (g/100 g sample) and 0.95, respectively, and slightly decreased upon storage. Frozen water content was 51% (g frozen water/100 g water sample) in fresh STD, 68% in P-W and 48% in P-STD and decreased significantly in all samples during storage to 46, 55 and 28%, respectively. Retrograded amylopectin increased in all samples and it was significantly reduced in P-W as compared to STD and P-STD. <sup>1</sup>H FID (up to 0.1 ms) was more mobile in P-W and less mobile in P-STD, as compared to STD. At day 7 STD e P-W were comparable. T<sub>2</sub> times distributions in fresh samples had three <sup>1</sup>H populations: pop A (0.5 ms; ~28% of relative abundance in STD, 25% in P-W and 30% in P-STD), pop B (8-10 ms; ~66% of relative abundance in STD, 69% in P-W and 64% in P-STD) and pop C (130 ms; ~5-6% of relative abundance in all samples). During storage, pop A remained almost constant in P-STD and P-W and decreased from 28 to 25% in STD. Pop B slightly increased in STD (from 66 to 69%) and slightly

decreased P-W (from 69 to 67%). Relaxation times of pop B decreased in all samples to 6-8 ms.



## DIFFERENT BEHAVIOR OF WATER IN FRUIT FILLINGS PREPARED WITH THE ADDITION OF INULIN, PECTIN AND GELLAN GUM

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In order to improve current fruit fillings and develop new ones with heat-stable properties, the basic understanding of water behavior with added hydrocolloids at the molecular level is required.

The main goal of this study was to analyze the structure and water-binding properties of fruit fillings within a wide range of soluble solids and added hydrocolloids, before and after freeze-thaw cycles, by using optical microscopy and low-resolution NMR techniques. The physical state of the water in fruit fillings is extremely affected by the initial water content, the amount of added carbohydrates, as well as the microstructure of the gel matrix formed. In order to characterize water-holding capacity of the fruit filling with different compositions including sol-gel transitions and syneresis, from the qualitative and the quantitative point of view, the LR-NMR by means of  $T_2$  relaxation times was applied. NMR  $T_2$  distribution revealed three distinct types of water: intra-colloid water inside gellan gum and pectin colloids, extra-colloid (capillary) and crystalline (structured) water that is highly linked to carbohydrates and found in high-soluble solids fillings. Data of the lowest  $T_2$  range, reflecting the lowest mobility of hydrogen protons, were strongly dependent on the total soluble solids and water content, whereas the highest spin-spin relaxation times (more mobile water fractions) displayed a slight increase in  $T_2$  when syneresis took place. According to the fact that the diffusive exchange of water molecules between intra- and extra-colloid water in fruit fillings is slow on the NMR timescale, the obtained relative intensity values of the analyzed relaxation spectra was taken as proportional to the water types and their localizations.

## **WATER CONTRIBUTION TO THE STRUCTURATION OF STARCH MATRICES IN THE PRESENCE OF FLAVOUR**

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Starch is able to form flavour-inclusion complexes with certain flavours via gelatinisation. This work focuses on the ability to form complexes under limited water and flavour interactions at different gelatinisation degrees. Wheat starch was used as the study model since it is widely used in biscuit and snack products. Literature search indicated that ethylhexanoate is able to form inclusion-complexes while 2-hexanone is unable. These two flavours are C-6 compounds with similar chemical properties, hence they were chosen for a comparison study. Application of heat-moisture treatment (65 and 85 °C) on two starch-water ratios (20/80 and 50/50 g of wet weight) brought about 0-32% relative residual crystallinity of the sample. No inclusion complex could be identified by DSC due low flavour concentration. However, the result of flavour analysis by simultaneous distillation extraction (Likens-Nickerson) and gas chromatography (GC-FID) analysis suggested that there was interaction between flavour and starch since flavours were detected. Starch pastes were freeze-dried to facilitate the study with Rapid Visco Analyser (RVA). Samples exhibited 0% relative residual crystallinity (by DSC results); however they still showed increasing viscosity upon heating and were thus totally different from pre-gelatinised starch. The overall results suggested that each recipe was different and the presence of flavour affected significantly their pasting profiles. RVA profiles emphasised the effect of heat-moisture treatment and limiting water as they involved changing gelatinisation profile of starch which cannot be identified by DSC. The complete result of this study is expected to be helpful for understanding the role of water in starch-flavour interactions.

## PHYSICOCHEMICAL CHARACTERISATION OF CARVACROL- $\beta$ -CYCLODEXTRIN INCLUSION COMPLEXES

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Carvacrol is a hydrophobic compound with recognized pharmacological activities. Cyclodextrins (CDs) are used to increase solubility and dissolution through water-insoluble compounds inclusion into their hydrophobic cavities. In this study, the inclusion complex of Carvacrol/ $\beta$ -CD was prepared by the slurry (SC) and paste (PM) methods. The formation of carvacrol/ $\beta$ -CD inclusion complex was demonstrated by different analytical techniques including differential scanning calorimetry (DSC) and thermogravimetry/derivate thermogravimetry (TG/DTG) and scanning electronic microscopy (SEM). Thus, the DSC curve of the PM indicates endothermic peaks which correspond to the release of water molecules as well as to the release of carvacrol, probably adsorbed to the surface. However, the DSC curve of the SC indicates only an endothermic event following decomposition. The difference in the DSC curves of the PM and the complex of carvacrol/ $\beta$ -CD clearly indicate complex formation between the components. The analysis of the TG/DTG curves evidences the complexation. The curves show in the range of 130-280 °C that the PM and SC lost 3.4% and 8.7%, respectively. This signifies that by SC, in this range carvacrol strong encapsulated is released, and at ~280°C, started the decomposition of  $\beta$ -CD molecules appears. The complexation between carvacrol and  $\beta$ -CD appeared as agglomerates. In contrast, the particle shapes and morphologies of the corresponding PMs were similar to those of  $\beta$ -CD. The drastic change of the particles' shapes and aspects in the SC sample were indicative of the presence of a new solid phase.

**SILYBIN, RUTIN AND THEIR FATTY ACID  
BIOCONJUGATE/CYCLODEXTRIN COMPLEXES AS POTENTIAL  
HEPATOPROTECTIVE AGENTS. A KARL FISHER WATER  
TITRATION APPROACH**

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Silybin occurring in *Silybum marianum* L. (milk thistle) is a well known hepatoprotective agent from the flavonolignan class, while rutin (*Carpobrotus edulis* L. - ice plant, *Ruta graveolens* L. - rue, and many other plants) is an antibacterial flavonol glycoside with potential hepatoprotective properties. Both natural flavonoid derivatives have antioxidant activities, but their bioavailability is low.

In the present study, the enhancement of the bioavailability of both silybin and rutin by two methods was performed. First, these potential hepatoprotective agents were encapsulated in natural cyclodextrins in order to enhance the water solubility [1]. Silybin and rutin were also enzymatically esterified with common fatty acids in order to enhance the cyclodextrin complexation properties. The molecular encapsulation efficiency was evaluated by means of water content of the corresponding complexes. The Karl Fischer water titration parameters were considered in order to discriminate between cyclodextrin complexed/uncomplexed as well as free/bioconjugated silybin and rutin.

[1] Hădărugă, D.I.; Hădărugă, N.G.; Bandur, G.; Isengard, H.-D., Water content of flavonoid/cyclodextrin nanoparticles: relationship with the structural descriptors of biologically active compounds, *Food Chemistry* **2012**, 132(4), 1651-1659.

## INFLUENCE OF FLAVONOID GLYCOSIDE NARINGIN AND HESPERIDIN FATTY ACID BIOCONJUGATION ON THE WATER CONTENT OF THEIR CYCLODEXTRIN COMPLEXES

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Naringin and hesperidin are flavonoid glycosides from citrus fruits such as grapefruit. It was demonstrated that the diet supplementation with these bioflavonoids conducts to a better insulin release and attenuate hyperglycemia. Furthermore, they have anti-inflammatory and anti-carcinogenic activities due to their antioxidant properties.

In the present study the enhancing of the bioavailability of these bioflavonoids having very low water solubility was performed by molecular encapsulation in cyclodextrins. In order to increase the capability of host-guest interaction, these bioflavonoids were derivatized to the corresponding fatty acid bioconjugates, which allows the cyclodextrin encapsulation due to the higher hydrophobicity and geometrically compatible molecules. Derivatization to the naringin- or hesperidin-fatty acid bioconjugates was achieved by enzymatic synthesis in anhydrous acetone. Bioconjugate-cyclodextrin complexation was performed by crystallisation from ethanol-water method. Naringin and hesperidin/cyclodextrin complexes as well as their fatty acid bioconjugate/cyclodextrin complexes were evaluated for the water content by Karl Fischer titration method, TG and DSC. The correlation between flavonoid or bioconjugate structures and water composition of the cyclodextrin complexes was established.

[1] Hădărugă, D.I.; Hădărugă, N.G.; Bandur, G.; Isengard, H.-D., Water content of flavonoid/cyclodextrin nanoparticles: relationship with the structural descriptors of biologically active compounds, *Food Chemistry* **2012**, 132(4), 1651-1659.

## ADSORPTION BEHAVIOR OF BULGUR

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Sorption isotherms are important in the food domain to specify conditions of storage, stability, packaging, drying, predicting shelf life and understanding the physico-chemical changes in the context of the production of dried foods. Bulgur is a traditional wheat product half-ready to eat. It is mostly produced from durum wheat by boiling, drying, dehulling, breaking, sifting and classifying. In this research, a gravimetric method was used to determine the adsorption behavior of bulgur. Three different bulgur samples differing in particle-size ( $2 < \text{coarse} < 3.5$  mm,  $1.6 < \text{medium} < 3.0$  mm,  $0.5 < \text{fine} < 2.0$  mm) produced from durum wheat and purchased on the market were kept in the atmosphere of eight saturated salt solutions with different relative humidity ( $\text{KC}_2\text{H}_3\text{O}_2$  22.5%,  $\text{MgCl}_2$  33.1%,  $\text{K}_2\text{CO}_3$  43.2%,  $\text{NaBr}$  57.6%,  $\text{KI}$  68.9%,  $\text{NaCl}$  77.5%,  $\text{BaCl}_2$  90.7% and  $\text{K}_2\text{SO}_4$  97.30%) at three different temperatures (20, 30 and 40 °C) for 7 days. For the determination of adsorption isotherms, the samples were dried to nearly 3% water content under vacuum at 40 °C. Samples of about 1 g in small glass dishes were put in desiccators and their changing weight was controlled every day to determine the time needed to reach the equilibrium. The obtained data were evaluated on the basis of two different sorption equations, BET and GAB. The sorption isotherms of bulgur samples were of type II (S-type is typical for polymers). This can be explained by the bulgur composition. It has a high content of polymers, namely starch and proteins. Depending on temperature and particle size, the BET equation constants  $C$  and  $m_0$  were determined to be 1.98 to 26.92 and 2.36 to 3.73%, respectively. The GAB equation constants  $C$ ,  $m_0$  and  $k$  were found to be 1.38 to 66.90, 1.16 to 6.19% and 0.15 to 0.91, respectively. The BET and GAB equations can be used to calculate for monolayer water content ( $m_0$ ) of bulgur, because the constants  $C$  were calculated between 1 and 200 and  $k$  lower than 1. The  $m_0$  values decreased with increasing temperature and decreasing particle size.

## SUPERHEATING STEAM COOKING TO ENHANCE FLAVOURS OF POTATOES

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**Introduction.** Steam cooking at atmospheric pressure is a cooking process in which the food is placed above a boiled water compartment. The vapor is formed at atmospheric pressure, i.e. at a temperature close to 100 °C. In this process, there is no direct contact between the food and the cooking water. Steamed vegetables are known to have higher nutritional qualities than those cooked by immersion in water. However, the foods cooked by this method are often described as "tasteless". This process operates in an open system, which causes the loss of a significant part of the aroma compounds (1). Cooking by superheated steam has been proposed in order to develop new methods for steam cooking of both the conservation of the nutritional and organoleptic qualities.

**Materials and methods.** The study was carried out with potatoes because they are the product most commonly steam-cooked and with a model matrix containing potato starch. Two kind of cooking processes were studied: 1) a steam cooking process at atmospheric pressure where potato or model matrix cylinder samples were cooked in a prototype cooker during 23 min and 2) a superheating steam cooking process composed of a steam generator and a super-heater providing steam at 145 °C.

The behavior of 6 aroma compounds known to have a strong impact on potato flavor (2), analyzed using gas chromatography, were compared for these two processes.

**Results.** Overall, the aroma compound contents were higher with superheated steam cooking for both the potatoes and the model matrix. With superheated steam cooking, a crust was formed on the surface of the food which acted as a barrier to the aroma compound release and minimized leaching effects observed in conventional steam cooking.

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## EFFECT OF WATER AND GLUTEN ON PHYSICO-CHEMICAL PROPERTIES AND STABILITY OF READY TO EAT SHELF-STABLE PASTA

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Ready to eat shelf stable pasta (RTE pasta) has been introduced in the market to satisfy consumers' request for convenient food. RTE pasta has been reported to be subjected, during storage, to physico-chemical changes similar to those observed in bread staling resulting in decreased acceptability. In this work the effect of water and gluten have been investigated as possible means to improve product shelf stability.

A semolina dry pasta (STD) and a 15% gluten enriched pasta (GLU) were cooked to 56% (STD-56) and 59% g H<sub>2</sub>O/100 g product (STD-59 and GLU-59), packed into retortable pouches, sterilized ( $F_0=7$ ), and stored at room temperature for 2 months. Hardness, fragility and water status (moisture and DSC frozen water contents, proton mobility by 20MHz <sup>1</sup>H NMR [FID, T<sub>2</sub> distribution]) were measured during storage.

Moisture and frozen water contents were constant during storage. STD-56 lost plasticity and became fragile after 30 days, while GLU-59 and STD-59 retained a flexible structure for 60 days. Hardness and retrograded amylopectin increased during storage in all samples, less significantly in STD-59 and GLU-59. During storage, <sup>1</sup>H FID became steeper in all samples. <sup>1</sup>H T<sub>2</sub> distributions had three proton populations (A, B and C relaxing at ~0.25, ~11, and ~35 ms, respectively) until 3 days and then populations A and B merged into a single broad population. The amount of protons of the most abundant population C increased during storage. <sup>1</sup>H T<sub>2</sub> relaxation time of population C shifted to shorter times during storage. Increased water and gluten content in RTE pasta had a positive effect in preserving product's quality during storage.

**EVALUATION OF THE EFFECT OF TWO ANTIOXIDANTS  
FORMULATIONS ON CHEMICAL, PHYSICAL AND  
MICROBIOLOGICAL PROPERTIES OF A KIWI JAM DURING  
TWENTY WEEKS OF STORAGE**

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The general objective of this work was the development of a Kiwi (*Actinidia deliciosa* variety 'Hayward') jam preserving the high natural ascorbic acid content of the fresh fruit, as well as achieving the texture, sweetness and acidity characteristics of jams.

The optimised basic formulation presented 45% of fruit, 40% of sugar and 0.5% of Amidated Low Methoxyl Pectin (LMA). Three assays were tested: formulation with citric acid (CAC), formulation with ascorbic acid (CAA) and a control (CC) without antioxidant additions. No other preservatives were added. A pasteurization step of 20 min at 100 °C was applied. Samples were stored at room temperature for 20 weeks. Sampling for all chemical, physical and microbiological parameters, was performed after production and at weeks 2, 4, 8, 12, 16 and 20. All samples were analysed in triplicate. SPSS statistics 17.0 was used for statistical analysis.

Moisture,  $a_w$ , ash, and total soluble solids contents were found to be constant in all samples along the storage period. The pH value decreased until week 2 and increased after that until week 20, whereas titrable acidity decreased gradually along storage in all formulations. No significant differences on the activity of antioxidants by DPPH was found in CAC samples compared to the control, but the antioxidant activity of CAA samples was significantly higher than the others. In all samples the ascorbic acid content gradually decreased during storage, and at the end losses were of 35%, 48% and 44% of the original ascorbic acid content, respectively, in formulations CAC,

CAA and CC. No significant differences in colour were detected during the storage period, but the texture of CAC samples was significantly different, decreasing gradually during storage. In all samples, throughout the storage period, microbiological counts (TVC, yeasts and moulds) were below the limit of detection.

**STUDY OF THE EFFECT OF PH ON COLOUR OF FLAVOURED SOLUTIONS OF *SPIRULINA SPP.* AND *CHLORELLA VULGARIS***

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Microalgae have been identified as an alternative and innovative source of nutrients considered important to human diet, in particularly as a source of natural antioxidants containing not only carotenoids but also polyphenols. The microalgae *Chlorella vulgaris* (Beijerinck) have been also widely studied for human consumption purposes not only because of their antioxidant properties but also as a protein-rich “health” food. *Spirulina* spp., which has been reclassified as a Cyanobacteria and renamed *Arthrospira* spp., has also been widely studied as a food ingredient or supplement. Preliminary works have shown that the stability of the characteristic green colour of *Chlorella vulgaris* is more susceptible to low pH and to heat treatments compared to the *Spirulina* spp. blue/green colour. The general purpose of this study is to use a mix of *Chlorella vulgaris* and *Spirulina* spp. as ingredients for the future development of a beverage product with high protein and antioxidants contents preserving the natural green colour considered to be an added value factor to the product. In order to evaluate the influence of pH on the stability of the colour of these microalgae, solutions of concentrated lemon juice (54 °Brix) with 0.10% of *Spirulina* spp. and 0.20% of *Chlorella vulgaris*, up to 100 ml of water, were prepared at three pH values: 9.08 (control without lemon juice), 4.58 and 4.32. The colour of the solutions was measured immediately after the preparation and after a thermal treatment (T=80 °C, 5 min). The following parameters were registered: lightness (L\*), colour coordinates a\* and b\*, saturation (c\*) and hue (h°).

The characteristic colour was maintained at all pH ranges at the end of preparation but after heat treatment only the solution at pH 9.08

preserved it. Although there was a slight improvement of colour stability, compared to previous works with *Chlorella vulgaris* alone, other mixes and concentrations of the two species must be tested to overtake the effect of the pasteurization critical step.

**P 14**

**THE ANTIMICROBIAL MECHANISM OF ELECTROLYZED  
OXIDIZING WATER AND ITS ROLE IN THE FOOD INDUSTRY.  
AN OVERVIEW**

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Food-borne diseases represent a significant threat to public health worldwide. They are much more of a concern to governments and food industry than a few years ago. The top categories of food linked to food poisoning are: seafood, dairy products, eggs, beef and poultry products. The best way to lower the incidence of food-borne diseases is to secure safe food supply. The development of effective methods for the reduction or elimination of pathogens in food and also in agricultural products represents an important issue in the food industry. Electrolyzed oxidizing water (EOW) has gained attention in the last years in the food industry for its efficiency in microbial inactivation. The main advantages of EOW are safety, reduced costs of production, low impact on the environment and consumers health. Studies are focused on the use of EOW for fresh-food industry and for its use as disinfectant in the food industry. Researches have shown its efficacy against pathogens commonly present in food. This review includes a brief overview of issues related to EOW mechanisms of action and its effective role in the food industry.

## PHTHALATES CONTAMINANTS IN BOTTLED WATER

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Phthalate esters are well known pollutants with potential risks for human health [1]. Different techniques such as liquid–liquid extraction and solid-phase extraction were widely employed for the determination of phthalate esters in water samples [2-6].

In this study, phthalate esters employed as plasticizers in PET bottles materials and their leaching into drinking water samples were investigated using liquid–liquid extraction techniques followed by gas chromatography–mass spectrometry (GC–MS) determination of the level of the target species (dimethyl phthalate, diethyl phthalate, di-*n*-butyl phthalate, benzyl-butyl-phthalate, ethylhexyl-phthalate and dioctyl-phthalate). Low levels of phthalate esters contaminants were thus identified in several commercially available Romanian bottled mineral water samples.

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**CARP MIOFIBRILAR PROTEIN CONCENTRATE DRY BY USING  
SPRAY DRYER TECHNOLOGY AND ELEMENTAL MAPPING OF  
MICROSTRUCTURES BY SCANNING ELECTRON MICROSCOPY**

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Fish protein ingredients production has a growing trend all around the world because of their low cost, high nutritive quality and more concentrated protein levels. The current study refers to miofibrillar protein extraction and their characterization of fresh carp meat. Acid dissolution, followed by precipitation at the isoelectric pH was used as extraction method. The miofibrillar protein chemical characterization was made by taking into account the functional and rheological properties. Spray-dryer method for miofibrillar proteins solubilized at alkaline pH and acid pH and scanning electron microscopy (SEM) were used for dry. The solubility of the muscle proteins, constituent components of protein derivatives, is a critical property that controls the other functional characteristics of the protein (emulsifying capacity, foaming and gel formation). The protein concentrates/isolates, studied by their functional properties, protein solubility and gelling characteristics, can be suitable raw materials for protein films and biodegradable coatings generation.



**INVESTIGATION OF WATER QUALITY FOR FOOD PROCESSING:  
MONITORING OF THE NITROSAMINES CONTENT BY ULTRA  
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY TANDEM  
MASS SPECTROMETRY**

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In the food industries, water is used during production and cleaning steps and also as a raw material. Thus, its quality is an important issue to guarantee the food security and safety. Generally, water for food processing should meet the existing standards for drinking water. Some compounds in water need to be strictly controlled, especially the emerging organic micropollutants which are well-known to affect the human health and most of them are still not regulated. Among them, nitrosamines are well known to be potential carcinogens and they have been detected at trace levels in water.

The aim of this study is to develop a fast, sensitive and efficient analytical method for the routine determination of low part per trillion levels of N-nitrosamines in drinking waters. An ultra high pressure liquid chromatography coupled with tandem mass spectrometry (UHPLC/MS/MS) method was developed for the qualitative and quantitative analysis of the selected compounds. Under optimized analytical conditions the separation of analytes is done in less than 2 minutes. The obtained limits of detection were between 0.08 and 0.32 ng L<sup>-1</sup>. The extraction recoveries were in the range of 81-100%.

**THE INFLUENCE OF THE STORAGE MICROCLIMATE ON THE FRESHNESS OF THE APPLES, DETECTED THE ELECTRONIC NOSE**

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Water represents the environment for the realization of biochemical reactions, taking part in the process of vehiculation of the dissolved substances and has a role in maintaining the fruit's turgor. Until harvesting, plant tissue stores water, the vital component, which is the slowly lost during the post-harvest period through the process of sweating. The process is more intense at the beginning of storage and gradually decreases as the content of dry matter increases, due to the hydrolysis of starch and soluble cellular juice concentration. Apples should be harvested before they are fully ripe, but giving them time to mature. Immature apples have a starchy taste, undeveloped aroma and are very hard and crisp. Sugars are major soluble solid in fruit juices and therefore soluble solids are often used as an estimate of sugar content. In this moment, also, respiration intensity is low and apples are characterized by a minimal respiration. Mature apples are firm but not hard. As the fruit mature, turgor diminishes, the starch changes to sugar and the aroma and flavors develops, when volatile compounds are produced. Emission of volatile components determines the increase of respiratory intensity, process at the stage of maturity of the apples reach at a maximum respiratory, respective maximum climacteric. The perception of volatile compounds by the human nose is of great importance in evaluating quality of foods; therefore, a similar principle as the human nose, the electronic nose, was used. An electronic nose (E-nose) was used to classify apple samples based on their smell, depending on the degree of maturation. Seven varieties of apples from Romania were examined. All the samples were analyzed using the E-nose FOX 4000 with 18 metal oxide coated or uncoated sensors. The resulting E-nose intensities were analyzed by principal component analysis (PCA), discriminant factor analysis (DFA) and statistical quality control (SQC), which resulted in grouping the used varieties of apples or in grouping the types of samples (peel,

homogenate or diluted homogenate from the same apple). The obtained results indicated that the E-nose could discriminate successfully among varieties of apples (% of variance  $\gg 90$ ; percentage of recognition  $\approx 100\%$ ) and can be helpful in determination of optimal storage period.

## WATER QUALITY USED IN THE TECHNOLOGICAL PROCESS OF OBTAINING BEER

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Water is the second main raw material, after malt, which profoundly influences the quality of the obtained beer, especially as it presents 80-90% of the final product. The quality of water used in brewing plays a very important role in the development and expression of the organoleptic characteristics of the beer.

SC Heineken Romania SA Brewery with its branch in Craiova monitors water quality by taking samples within its own program of production, but samples are also analyzed in the "Aware of Water" laboratory of Heineken in the Netherlands and by Surveillance Program DSVSA Control and Dolj.

Such detailed analysis of the water used for brewing Heineken beer in Craiova comprises a total of 290 parameters, namely: 29 physico-chemical, 19 metals, 6 products resulting from water disinfection, 42 aromatic hydrocarbons, 25 volatile organochlorine compounds, 15 polycyclic aromatic hydrocarbons (PAH), 11 other volatile compounds, 12 alkanes and mineral oil fractions, 3 phthalates, 28 phenols, 7 biphenylpolychloride compounds (PCB), 40 organochlorine pesticides, 44 organophosphorus pesticides.

In this paper physico-chemical analyses are presented, the content of heavy metals, PCBs and organochlorine and organophosphorus pesticides in the water used for brewing Heineken EU in comparison with Directive 98/83/EC and compared with the standard set by Heineken. From the physico-chemical point of view the 29 parameters analyzed were below the EU standards, as well as under the standard set by Heineken.

The content of heavy metals is also under the EU and Heineken standard, higher values occurring in the case of iron.

The concentration of PCBs in the 8 parameters analyzed revealed that all of them were below EU and Heineken standards.

Organochlorine pesticide residues reveal that all the 40 parameters analyzed are below national standards and the EU and Heineken standards as well.

## COMMON-ION EFFECTS ON DELIQUESCENCE LOWERING OF CRYSTALLINE INGREDIENT BLENDS

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Deliquescence is a first-order phase transformation of a crystalline solid to a saturated solution that is initiated at a defined relative humidity (RH),  $RH_0$ . The presence of multiple deliquescent solids in a blend lowers the deliquescence point (denoted  $RH_{0mix}$  for blends). Deliquescence RHs have been determined by measuring the  $a_w$  of a saturated solution, as well as by water sorption techniques. The Ross equation has been used to predict the  $RH_{0mix}$  in a blend by multiplying the individual  $RH_0$ s of the ingredients of interest. For many blends, the Ross equation prediction of  $RH_{0mix}$  is accurate; however, unexplained larger variations have been found in some blends. This study investigated the effects of common ions (both anions and cations) in ingredient blends on the measured  $RH_{0mix}$  and compared these values to the Ross equation predictions. The deliquescence points of common ionic food ingredients (NaCl, KCl, ascorbates, thiamine HCl, and citrates) and blends thereof (equal ingredient ratios) were determined using water activity ( $a_w$ ) and DDI (Decagon Aquasorb) measurements. In binary blends with no shared ion, the greatest deviation between predicted and measured  $RH_{0mix}$  was 8% RH lower (enhanced solubility) and 2% RH higher. When an ion was shared in a binary blend, the measured  $RH_{0mix}$  was consistently 7-8% RH higher than the Ross equation predictions, and the  $RH_{0mix}$  of tertiary blends with a common ion were 16-18% RH higher than predicted. The diminished deliquescence lowering (higher than predicted  $RH_{0mix}$ ) caused by the common-ion effect can be explained by Le Chatelier's principle: the common-ion will compete while going into solution. At equilibrium, the solution will be saturated with the common-ion while the counterions' solubility will decrease due to the inability to dissociate at the same concentration. With an overall decrease in solubility, there will be fewer dissociated ions in the solution and the vapor pressure will be higher, as explained by Raoult's law. Since the  $a_w$  of a saturated solution is the

same as the deliquescence point, the  $RH_{0mix}$  is increased due to the decrease in solubility of the blend when an ion is shared between the ingredients.

## MEASURING WATER VAPOR PERMEATION FOR FOOD PACKAGE MATERIALS USING CRDS

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One of the critical factors in the choice of food packaging materials is moisture permeability which is important for maintaining product quality. Various types of barrier-coated polymer films are being used to package many different food products. A continuing trend of food packaging is to extend the shelf life of food while maintaining fresh-like quality.

KRISS has been operating a measurement system for moisture permeation using water containing tritium since 2009. Its detection limit reaches to  $10^{-7}$  g/m<sup>2</sup>day. However, it is difficult to utilize it for general purpose because tritium is a radioactive isotope.

Therefore, we have established another method for measuring water vapor permeation in a range of  $10^2$  to  $10^{-5}$  g/m<sup>2</sup>day by using cavity ring-down spectroscopy (CRDS). The accuracy is improved by a low-flow gas feedback circuit with low-pressure and by controlling the adsorption/desorption periodically. This method provides absolute quantitative measurement, which has a traceability to national standards. The uncertainty of the CRDS-based measurement device was about  $1 \times 10^{-5}$  [g/m<sup>2</sup>day].



**INVESTIGATION OF WATER FEATURES OF BISCUIT DURING STORAGE THROUGH THE USING A RAPID DYNAMIC DEWPOINT METHOD**

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Quality preservation of complex multi-domain foods, such as biscuits, is a very complicated issue because of the different chemico-physical phenomena taking place simultaneously during storage. Although dry biscuits are considered to have a relatively long shelf-life, due to their low water activity, the type and entity of the interaction between their solid matrix and water fraction can change during storage, being the main responsible of quality deterioration phenomena.

The objective of this study was to investigate the water sorption behaviour of biscuits during accelerated storage. Adsorption isotherms of commercial biscuit samples were obtained using a rapid dynamic dewpoint method (DDIs) during 92 days of accelerated storage (35 °C, 50% RH). During storage samples were also analysed for water content, water activity and peroxide value.

GAB and BET models were used to fit the experimental sorption behaviour. With the passing of storage time, the equilibrium water content of the biscuits, at a given water activity, increased in the 0.03-0.4  $a_w$  range and obtained isotherms underwent to modification of behaviour and shape.

Moreover, the monolayer moisture content calculated with the BET equation increased from 1.473 to 2.080 g per 100 g  $dw^{-1}$ , from the beginning to the end of storage.

These results could be ascribed to an increase of water sorption polar sites, as a consequence of chemical and physical changes of sugar and biopolymers (e.g. starch and protein) induced by product ageing.

## INFLUENCE OF DIFFERENT DRYING METHODS ON THE PHYSICOCHEMICAL PROPERTIES OF PUMPKIN

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This study was carried out to determine the influence of hot air drying and combined methods on physicochemical properties of pumpkin (*Cucurbita moschata*) samples. The experiments in the hot air chamber were executed at 50, 60 and 70 °C.

The combined method consists of a triple combination of the main drying techniques. Thus, in first stage the samples were dried by hot air convection at 60 °C followed by hot air ventilation at 40 °C simultaneously with microwave heating (microwave power of 105-315 W).

The drying curve was analyzed to determine the drying time, moisture content and rehydration capacity.

The time required to reduce the moisture content to any given level was highly dependent on the drying conditions. So, the highest value of drying time in hot air was 540 min at 50 °C, while the lowest time was 189 min in hot air combined by microwave heating at 40 °C and a power of 315 W.

The samples dried by hot air show a higher rehydration capacity (92.92 - 79.81%) than samples dried by the combined method (87.05 - 77.68%).

## THE INFLUENCE OF PHOSPHOLIPASES ON THE WATER CONTENT OF VARIOUS BREAD TYPES

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Enzymes are often used in the obtaining of baked goods, bakery and bread products in order to hydrolyze the triglycerides, polar lipids (such as glycolipids), and phospholipids. The phospholipid hydrolysis is important for the overall surface tension due to the splitting of the fatty acid groups from the phospholipid structure. As a result, all physico-chemical characteristics of these food products are influenced, including the water content and the type of water molecules which are retained in the food matrix.

In this study the influence of using phospholipases as food additive in obtaining of bread products on the water content and type of water molecules was evaluated. Thus, various concentrations of phospholipase and types of flour (wheat flour, whole meal flour, Graham flour and rye flour) were used in order to obtain bread samples. Among the other physico-chemical parameters (acid index, fatty acid content, phospholipid content etc.), the water content and the type of water molecules (i.e. "surface" or "strong-bounded") were evaluated; the Karl Fischer volumetric titration method was used for determining the water concentration and types of molecules from bread samples. The water content and type of water molecules, as well as the other physico-chemical parameters of the phospholipase-containing bread samples were correlated with the concentration of enzymes used as food additive.

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**A STUDY REGARDING PRESERVATION PERIOD OF A DIETETIC MEAT PRODUCT**

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In this study a dietetic minced meat product was obtained by total replacement of animal fat by a vegetable oil mixture. Extra virgin olive oil and palm oil were the two olive types used. They are high in unsaturated fatty acids, vitamins and lecithins and low in cholesterol. The benefic presence of unsaturated fatty acids regarding human health implies the technological risk of a faster oxidation comparing to a standard product and thus a lower shelf life. There is a strong connection between water activity and microbiological growth. In order to observe the influence of vegetable oil in meat on water activity and microbiological load, results were compared with a standard product in which animal fat was used.

Knowing the value of water activity can be a good guidance for a product's preservation time. Studies from the domain suggest that in order to avoid any microbiological risk, it takes a water activity value less than 0.7.

The water activity immediately after the technological process was 0.965 and decreased after 14 days to 0.920 for the dietetic product and to 0.966 and 0.932 respectively for the control product with animal fat in its composition. However microbiological results showed a low bacterial load for the fresh dietetic product (12 ufc) and a much higher load for the control one (36 ufc). Our results showed that the new dietetic product obtained by replacing animal fat with vegetable oils had a reasonable preservation period for the meat product category to which it belongs.

**WATER CONTENT OF TRITERPENOID SAPONINS AND THEIR  
FATTY ACID BIOCONJUGATE/CYCLODEXTRIN COMPLEXES**

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Triterpenoid glycoside saponins are a very important phytochemical class with implications in both food and pharmaceutical fields.

The triterpenoid glycoside saponin concentrates extracted from liquorice root (*Glycyrrhiza glabra* L.) containing glycyrrhizin, horse chestnut (*Aesculus hippocastanum* L.) containing  $\beta$ -aescin, and ginseng root (*Panax ginseng* L.) containing ginsenoside were complexed in  $\beta$ - and  $\gamma$ -cyclodextrin. Furthermore, the main specified bioactive compounds were enzymatically esterified to the corresponding essential fatty acid bioconjugates by using Novozyme 435 in anhydrous acetone. The resulting triterpenoid saponin-fatty acid bioconjugates / cyclodextrin complexes were also synthesized by using the crystallisation from ethanol-water solution method. Both triterpenoid saponin/cyclodextrin and triterpenoid saponin-fatty acid bioconjugate/cyclodextrin complexes were analyzed for the water content by using bi-component Karl Fischer titration and thermogravimetry. The enhancement of cyclodextrin molecular encapsulation of triterpenoid saponin esters in comparison with the raw bioactive compounds was emphasized by means of the Karl Fischer water titration parameters.

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## **EXHIBITION STANDS**







## **EXHIBITION STANDS**

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