

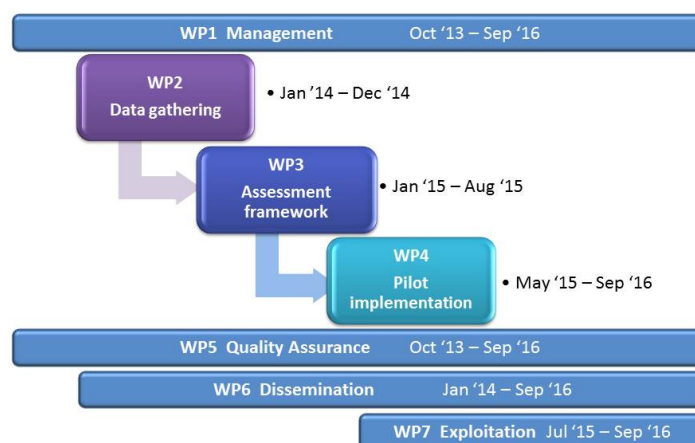


## Improving Teaching Effectiveness in Chemical Engineering Education

This consortium brings together six European academic institutions (including partners from the UK, France, Republic of Macedonia, Portugal, Slovakia and Germany) providing chemical engineering degrees with professional / accreditation bodies and employer organisation representatives (as associate partners of the consortium). The aim of the project is to develop a framework which will support the assessment of teaching effectiveness in delivering not only core chemical engineering knowledge, but also core employability competencies.

The project officially started on the 1<sup>st</sup> October 2013 and will complete its activities on 30<sup>th</sup> September 2016. It is divided into seven work packages.

### Project overview



Work packages 2, 3 and 4 will deliver the aims of the project through the following objectives:

- Review the learning outcomes of a chemical engineering higher education formation (as set out by the Bologna recommendations and professional accreditation requirements), in consultation between industrial and academic partners. (WP2)
- Promote closer involvement of employer organisations in chemical engineering curriculum formation by carrying out focus groups and semi-structured interviews with associated industrial network partners, as well as questionnaire surveys of wider labour market representatives, to identify the skill gaps and requirements. (WP2)
- Establish state-of-the art in assessing the effectiveness of teaching of core (chemical) engineering knowledge and of the development of professional skills and competencies required to increase the employability of the graduates. (WP2)
- Define various indicators of the effectiveness of teaching in chemical engineering higher education, share them with the community to help HE institutions to improve their formations both in chemical engineering as well as broader subject areas. (WP3)

- Investigate in more depth methods of effectively acquiring employability competencies, using psychometric approaches amongst others. (WP3)
- Use decision making technology and multiobjective optimisation to identify the most appropriate evaluation methods and develop a robust framework for supporting effective delivery of core knowledge and employability competencies. (WP3)
- Test the framework at partner institutions focusing on various pedagogic methodologies (e.g. recorded instruction material used for directed learning, problem based learning, work based learning, tutorial and traditional lecture delivery as well as practical laboratory instruction) in each geographical area to enable the investigation of dependencies between educational systems and the effectiveness of pedagogic methodologies. (WP4)

The consortium is keen to engage as many higher education chemical engineering degree providers and potential employers as possible. Get in touch with us through our website (<http://www.iteach-chemeng.eu>) or e-mail the project coordinator, Dr Jarka Glassey directly ([jarka.glassey@ncl.ac.uk](mailto:jarka.glassey@ncl.ac.uk)).

**Verka Meshko**

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**DOCTORAL THESES****DEFENDED AT THE FACULTY OF NATURAL SCIENCES AND MATHEMATICS,  
Ss. CYRIL AND METHODIUS UNIVERSITY IN SKOPJE, 2013****1****Marina Stojanovska****THE OCCURRENCE OF ERRONEOUS NOTIONS IN TEXTBOOKS AND CHEMISTRY  
TEACHING IN MACEDONIA AND POSSIBILITY FOR THEIR ELIMINATION  
(27.2.2013)**

A research concerning the presence of some erroneous notions (misconceptions) among primary- and high-school students was conducted in this thesis. The study deals with misconceptions from different chemistry areas and attention was paid to the misunderstandings related to the vague ideas of the relationship between the macro- and micro-world. An attempt was made to identify the origin of these misconceptions, to state the possibilities for their correction as well as to give some suggestions for progress in the chemistry teaching.

The purpose of this study was to address and face the potential misunderstandings among students and to prepare suitable intervention programs to fight the misconceptions. Intervention programs consisted of laboratory activities, animations, molecular models, video materials and discussions.

Qualitative methods, such as interviewing techniques, were applied and were very useful in gaining in-depth notions about students' knowledge. The research also involved questionnaires (chemistry concept inventories in a pre-test–post-test design) that were used to follow the improvement of students. The collected data were further analyzed using the software package Predictive Analytics SoftWare (PASW) 18.0.

The result of the study was that many misconceptions were detected, but, fortunately, they were reduced after the conducted intervention programs. It was found that the results in the post-test were significantly higher than those in the pre-tests which proved that the intervention programs were efficient, applicable and appropriate for teaching. However, a few misconceptions were still present in the post-tests, thus confirming the existence of difficulties in the process of ironing-out mis-conceptions.

We honestly hope that this research will be of great help for all present and future chemistry teachers in their lesson planning and preparing effective tests for their students. Surely, the textbook authors could also benefit from this research and make some interventions in order to present the material more clearly and more precisely where necessary.

*Keywords:* chemistry teaching, misconceptions, chemical reactions, symbolical representations, particulate nature of matter, oxidation reactions of monosaccharides, sublimation, liquid iodine, spectator ions.

**2****Katerina Bačeva****DISTRIBUTION OF HEAVY METALS IN ENVIRONMENTAL SAMPLES  
IN THE ALLCHAR MINE AND ITS ENVIRON  
(8.10.2013)**

The results from the investigation of the anthropogenic and geogenic influence of the presence of heavy metals in the Allchar locality, Republic of Macedonia, are presented in this PhD thesis, as well as characterization of the possible influence to the human health and living organisms

by the pollution with some toxic elements. The region's wider area, which is of interest in this investigation, is characterized by complex volcanic occurrences within the Kožuf massif in Vardar's geological zone. Although a number of geological and mineralogical investigations on the Allchar's region have been made, there have not been any detailed investigations of As, Sb, Tl and other toxic metals' possible influence over the pollution of the environment. This investigation includes the analysis of a significant number of soil samples, river sediments, water samples, and for the investigation of the air pollution, certain species of moss, and various floral endemic species from the investigated region have been used. During this investigation, 62 elements were analyzed (Ag, Al, As, Ba, Be, Bi, Br, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, I, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Rh, S, Sb, Sc, Sm, Sn, Sr, Ta, Tb, Te, Th, Tl, Tm, V, W, Y, Yb, Zn and Zr) in soil, water, sediments and moss samples. In order to achieve this, atomic emission spectrometry with inductively coupled plasma (AES-ICP) and mass spectrometry with inductively coupled plasma (ICP-MS) were applied. The obtained results were processed with specialized statistical analysis software and corresponding distribution maps were developed for various sampling media for each investigated element.

The results from the soil survey were processed by the method of multivariate factor analysis and six associations of elements were determined: Rb-Ta-K-Nb-Ga-Sn-Ba-Bi-Li-Be-(La-Eu)-Hf-Zr-Zn-In-Pd-Ag-Pt-Mg; Tl-As-Sb-Hg; Te-S-Ag-Pt-Al-Sc-(Gd-Lu)-Y; Fe-Cu-V-Ge-Co-In; Pd-Zr-Hf-W-Be and Ni-Mn-Co-Cr-Mg. For all analyzed elements and geochemical associations, distribution maps were prepared. The geochemical association of the As, Sb and Tl indicates mainly natural enrichment, but some high-values for Sb were result of former mining activity in the studied area. Anthropogenic influence to the distribution of Sb is also reflected in the river sediments as well.

Atmospheric pollution with certain toxic and heavy elements was measured using the moss biomonitoring technique in Allchar and its environ. By application of multivariate cluster and R-mode factor analysis, five geochemical associations were determined: F1 (Co, Cr, Fe, Sc, Li, V, Ga, Y, Ni, Mn, Al, La-Lu, Cu, Ge, Be, Bi and Hf); F2 (As, Tl, Sb and Mg); F3 (Rb, Cs and Mo); F4 (Sr, Ba, Hf, Zr, La-Lu and Bi) and F5 (Cd, Zn, Ag and Cu). The data from the samples around the Allchar mine and the rest of the Allchar area show that their source is mainly from natural phenomena. The comparison between the results from the samples around the Allchar mine and the rest of the Allchar area shows highest median values for As, Sb and Tl in the vicinity of the mine.

Within this thesis, an investigation for establishing the accumulation of some toxic elements in relation to its mobility in some local endemic plant species from this locality, *Viola allchariensis* G. Beck, *Viola arsenica* G. Beck, *Viola macedonica* Boiss. & Heldr., *Onobrychis degenii* Dörfler, *Thymus alsarensis* Ronn. and *Centaurea leucomalla* Bornm, was also carried out. The results showed that all studied endemic species are able to hyperaccumulate heavy metals in their roots, stems, leaves, flowers and seeds.

**Keywords:** Allchar, heavy metals, trace elements, soil, air, endemic species, pollution, water, sediments, moss

### 3

#### Lenče Velkovska Markovska

#### HPLC METHODS DEVELOPMENT FOR QUANTITATIVE DETERMINATION OF SOME HERBICIDES AND ORGANOPHOSPHATE INSECTICIDES IN VARIOUS MATRICES (9.10.2013)

The research presented in this work is contribution in the field of new analytical methods for identification and quantification of some herbicides (2,4-D and atrazine) and organophosphorus insecticides (malathion, parathion and fenitrothion) and their residues in various matrices. The development of relatively fast, simple, precise and accurate HPLC methods using ultraviolet diode array detector (UV-DAD) for the determination of pesticide residues in apple juice and water sam-

ples are explored. Furthermore, the new HPLC methods for quantitative determination of the active ingredients 2,4-D and malathion in the pesticide formulations Monosan Herbi, DMA-6 and Etiol tečni are developed.

For development of reversed-phase liquid chromatographic methods five analytical columns with different dimensions and particle size are used (LiChrospher 60 RP-select B (125 mm x 4 mm; 5 µm), LiChrospher 60 RP-select B (250 mm x 4 mm; 5 µm), Purospher STAR RP-8e (30 mm x 4 mm; 3 µm), Purospher STAR RP-18e (30 mm x 4 mm; 3 µm) and Poroshell 120 EC-C18 (50 mm x 3 mm; 2,7 µm)), while the developing of the normal-phase chromatographic methods is carried out using the intermediate column LiChrosorb CN (250 mm x 4 mm; 5 µm).

For quantitative determination of the active ingredient 2,4-D in the pesticide formulations Monosan Herbi and DMA-6, four reversed-phase (RP) liquid chromatographic methods are developed. Four RP-HPLC methods and one NP-HPLC method are proposed for determination of the active ingredient malathion in the pesticide formulation Etiol tečni. Five RP-HPLC methods for determination of residues of atrazine, malathion, parathion and fenitrothion in apple juice samples, and for determination of residues of 2,4-D, atrazine, malathion, parathion and fenitrothion in water samples, and one NP-HPLC method for determination of residues of parathion, malathion and fenitrothion in apple juice samples are developed. For enrichment and clean-up of compounds of interest Supelclean ENVI-18 SPE tubes were used.

The optimal conditions for separation and quantitative determination of the analytes with isocratic elution are obtained using mobile phases consisting of different ratios of acetonitrile/water in reversed-phase mode and n-hexane/dichloromethane in normal-phase mode, flow rate of 1 ml/min, constant column temperature of 25 °C and UV detection at 220 nm and 270 nm.

To determine the most effective procedure for the extraction of the investigated pesticides from water samples several organic solvents are tested: dichloromethane, a mixture of n-hexane-acetone (4:1, V/V), diethyl ether and petrolbenzene.

The validation of the developed methods is performed by testing the following parameters: specificity, selectivity, linearity, precision, accuracy, limit of detection and limit of quantification. Retention and separation factor, resolution, coefficients of determination, relative standard deviation of the results for retention time, the peak area and peak height, recovery of analytes and active ingredients quantity in the pesticide formulations, apple juice and water samples for each method are determined.

*Keywords:* 2,4-D, atrazine, malathion, fenitrothion, parathion, apple juice, water samples, Monosan Herbi, DMA-6, Etiol tečni, RP-HPLC, NP-HPLC, UV-DAD

#### 4

**Gjorgji Petruševski**

### **PSEUDOPOLYMORPHISM AND SOLID-STATE STABILITY OF SOME OPIATE ALKALOIDS STUDIED BY VIBRATIONAL SPECTROSCOPY AND THERMAL ANALYSIS (29.11.2013)**

The knowledge of the pseudopolymorphism and solid-state stability of each active pharmaceutical ingredient (API) is of crucial importance for selection of the most appropriate technological procedure for production of final pharmaceutical dosage form (further enabling its safe and efficient medical application). The PhD thesis comprises the results from the first detailed study related to the polymorphism of the commercial crystal hydrate forms of the narcotic-analgesic codeine phosphate. The phase transition interconversion pathway of codeine phosphate hemihydrate to sesquihydrate form, and vice versa, are thoroughly described. The obtained vibrational spectra and the results derived from the thermal analysis conducted on both studied API forms are elaborated. The existence of the metastable monohydrate and anhydrous form of codeine phosphate is also confirmed. Furthermore, solvatomorphism study governed by crystallization of the commercial form of

codeine phosphate sesquihydrate from various organic solvents shows that sesquihydrate undergoes direct phase transition either to hemihydrate (by crystallization from dimethylformamide), or anhydrous form. The latter outcome occurs when absolute ethanol, acetone and ethyl acetate are used as crystallizing solvents. In addition, a formation of solvate in methanol solution is also observed. The new obtained compound was identified as codeine phosphate sesquihydrate methanolate by means of various solid-state instrumental techniques. Corresponding solid-state study is conducted for pholcodine monohydrate. The work revealed that after crystallization from various organic solvents, pholcodine monohydrate exhibits unique crystal modification identical to the commercial form. In addition, single-crystal X-ray diffraction on specimen obtained from acetone solution, enabled to solve the crystal structure of pholcodine monohydrate. In the determined structure, each water molecule is connected by two pholcodine molecules via three strong hydrogen bonds. The obtained results offer new prospects towards solid-state properties of pholcodine monohydrate that might result in extended medical application of this important API.

*Keywords:* codeine phosphate, pholcodine monohydrate, solvatomorphism, polymorphism, vibrational spectroscopy, thermal analyses, X-ray diffraction, crystal structure

## МАГИСТЕРСКИ ТРУДОВИ

### ОДБРАНЕТИ НА ИНСТИТУТОТ ЗА ХЕМИЈА ПРИ ПРИРОДНО-МАТЕМАТИЧКИОТ ФАКУЛТЕТ, УНИВЕРЗИТЕТ „СВ. КИРИЛ И МЕТОДИЈ“ ВО СКОПЈЕ, ВО 2013 ГОДИНА

1. **Ивана Вучковиќ**, Следење на атмосферската депозиција на тешки метали во Република Хрватска со примена на биомониторинг со мов, атомска емисиона спектрометрија со индуктивно спрегната плазма и атомска апсорпциона спектрометрија.  
**Ivana Vučković**, Atmospheric deposition of heavy metals in Croatia studied by using moss biomonitoring technique, atomic emission spectrometry with inductively coupled plasma and atomic absorption spectrometry. (12.2.2013)
2. **Христина Томовска**, Разработка на GC-MS методи за анализа на изомерите на хексахлороциклохексан со различни начини на инјектирање.  
**Hristina Tomovska**, Comparison of various injection models in the GC-MS analyses of hexachlorocyclohexane isomers. (15.2.2013)
3. **Лина Анчева**, Квантитативна анализа на пептиди преку следење на специфични реакции на фрагментација со масен спектрометар со троен квадрупол.  
**Lina Ančeva**, Selected reaction monitoring triple quadrupole mass spectrometric targeted peptide quantification analysis. (8.5.2013)
4. **Александар Малетиќ**, Развој и примена на гасна-хроматографска метода за определување на фталатни естери од европската приоритетна листа во природни води.  
**Aleksandar Maletić**, Development and application of a gas chromatographic method for determination of phthalate esters from the European priority list in natural waters. (10.5.2013)
5. **Буран Скендер**, Разработка на метода за анализа на гликоли во почва со гасна хроматографија со примена на дериватизација.  
**Buran Skender**, Development of a method for analysis of glycols in soil by gas chromatography with derivatization. (11.7.2013)
6. **Емилија Колс**, Слаби интермолекуларни интеракции во втечнети инертни гасови. Теориска студија со комбинирана примена на статистичко-физички и квантномеханички пристап.  
**Emilija Kols**, Weak intermolecular interactions in liquid inert gases. A theoretical

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7. **Сузана Лазаревска**, Биохемиски анализи важни за следење на пациенти со шеќерна болест тип 2, кои се со аплицирана орална терапија.  
**Suzana Lazarevska**, Biochemical analyses important for monitoring diabetes type 2 patients with applied oral therapy. (10.10.2013)
8. **Филип Свонсон**, Вибрациони спектри на дикаезиум калциум тетрачлорид ди-хидрат и константи на анхармоничност за изотопски изолираните изотопомери на молекулите вода кај некои метални хлориди кристалохидрати.  
**Filip Svonson**, Vibrational spectra of dicaesium calcium tetrachloride dihydrate and constants of anharmonicity of isotropically isolated isotopomers of water molecules in some metal chloride crystallohydrates. (08.10.2013)
9. **Антонио Буџаревски**, Синтеза и структурна карактеризација на нови бензамидометил деривати на метансулфонамидот со потенцијална биолошка активност.  
**Antonio Bužarevski**, Synthesis and structural characterization of new benzamidomethyl derivatives of methan-sulfonamide with potential biological activity. (13.11.2013)
10. **Јасна Темелкоска**, Проучување на можноста за примена на хаотропни ре-агенси за анализа на опијатни алкалоиди и амфетамини со реверзно-фазна високоефикасна течна хроматографија.  
**Jasna Temelkoska**, Studying the possibility of using chaotropic reagents for reversed-phase HPLC separation of selected opiate alkaloids and amphetamines. (15.11.2013)
11. **Јелена Михајловиќ**, Разработка на UV-VIS спектрофотометриски метод за определување на сулфаметоксазол и сулфаметрол во фармацевтски и ветеринарни формулации.  
**Jelena Mihajlović**, Development of UV-VIS spectrophotometric method for the determination of sulfamethoxazole and sulfametrol in pharmaceutical and veterinary formulations. (10.12.2013)
12. **Каролина Русевска**, Разработка на метода за детектирање на егзогени супстанции во коса со гасно-масена спектрометрија.  
**Karolina Rusevska**, Development of method for detection of exogeneous substances in hair using gas chromatography coupled to mass spectrometry. (11.12.2013)
13. **Татјана Мицевска**, Разработка на модел за пресметување на октански број на бензини преку експериментални дескриптори.  
**Tatjana Micevska**, Newly developed model for calculating the octane number of gasoline using experimentally obtained descriptors. (13.12.2013)
14. **Моника Радевска**, Нов пристап кон спектроскопски и структурни испитувања на бубрежни камења.  
**Monika Radevska**, New approach for spectroscopic and structural characterization of urinary calculi. (17.12.2013)
15. **Александар Маркозанов**, Испитувања на различни видови мастила со помош на микро-раманска и инфрацрвена спектрографија и хеометриска анализа.  
**Aleksandar Markozanov**, Study of different types of inks using micro-Raman and IR spectroscopy and chemometrics. (18.12.2013)
16. **Јованка Илиева**, Разработка на метода за анализа на етеричното масло и липидната фракција од јатката на плодот на *Myristica fragrans* Houtt. со гасна хроматографија спрегната со масена спектрометрија.  
**Jovanka Ilieva**, Development of method for analysis of essential oil and lipid fraction from the kernel of *Myristica fragrans* Houtt by gas chromatography coupled with mass spectrometry. (11.06.2013)
17. **Наталија Крушаровска**, Разработка на метода за определување на автентичноста на млечни производи преку одредување на масно киселинскиот профил.  
**Natalija Krušarovska**, Development of a method for establishing the authenticity of dairy products by determination of fatty acid profile. (12.6.2013)
18. **Сања Милковска**, Испитување на механизмите на гликација на моделни пептиди.  
**Sanja Milkovska**, Investigation of model peptides glycation mechanisms. (22.2.2013)

**ДИПЛОМИРАНИ СТУДЕНТИ****НА ИНСТИТУТОТ ЗА ХЕМИЈА ПРИ ПРИРОДНО-МАТЕМАТИЧКИОТ ФАКУЛТЕТ,  
УНИВЕРЗИТЕТ „СВ. КИРИЛ И МЕТОДИЈ“ ВО СКОПЈЕ, ВО 2013 ГОДИНА****Применета хемија****Модул: аналитичка биохемија**

Начева Иле Љупка, 29.1.2013  
Димитровски Тони Ненад, 30.1.2013  
Филоvsка Јовица Катерина, 18.2.2013  
Ристоска Крсте Даринка, 8.3.2013  
Јордановска Ампе Марина, 19.3.2013  
Трајкоска Гоце Мирослава, 25.3.2013  
Димитрова Венцислав Тања, 25.3.2013  
Анчевска Симе Елена, 5.4.2013  
Башеска Сашо Викторија, 26.4.2013  
Колевска Зоран Милена, 9.5.2013  
Методијева Зоранчо Илина, 19.6.2013  
Рунчевска Миле Катерина, 20.6.2013  
Талевска Љупчо Лена, 1.7.2013  
Стојановска Блажо Моника, 5.7.2013  
Чергарска Михаил Катерина, 8.7.2013  
Ѓореска Веле Андријана, 10.7.2013  
Трајковиќ Радован Стефан, 12.7.2013  
Цекова Зоран Елена, 5.9.2013  
Зафировски Борче Благица, 13.9.2013  
Богданоска Коце Мирјана, 19.9.2013  
Златановска Бранко Марина, 19.9.2013  
Перевска Мите Ружица, 19.9.2013  
Кузманоска Душан Анита, 19.9.2013  
Камчевска Вангел Благица, 30.9.2013

Богоевска Благој Ивана, 9.10.2013

Спахиќ Фикрет Санела, 14.11.2013

Стојановска Лазо Маја, 30.12.2013

**Применета хемија**

Димитриеска Илија Јелена, 7.3.2013  
Зекири Адем Исен, 28.3.2013  
Ѓорѓиеvsка Пецо Ангелина, 5.4.2013  
Андов Бранко Ѓоре, 8.4.2013  
Кусевска Слободан Елена, 27.8.2013  
Јовановски Стојанче Стефан, 13.9.2013  
Васовиќ Божо Драгана, 23.9.2013  
Смилеска Славе Бисера, 9.10.2013  
Христовска Ѓорѓи Мила, 10.10.2013  
Крстева Миле Марија, 11.12.2013

**Наставна насока**

Манческа Димитрија Христина, 8.4.2013  
Јанковски Миле Јанко, 20.5.2013  
Србиноска Љубан Снежана, 11.7.2013  
Попеска Спасе Марија, 23.9.2013  
Јовановска Милослав Анета, 09.10.2013  
Ефтимова Петре Марија, 09.10.2013  
Љубенска Слободан Билјана, 10.10.2013



## DOCTORAL THESES

DEFENDED AT THE FACULTY OF TECHNOLOGY AND METALLURGY,  
Ss. CYRIL AND METHODIUS UNIVERSITY IN SKOPJE, 2013

1

Elena Veličkova

**NON-THERMAL PRESERVATION OF FRUITS AND VEGETABLES**  
**(30. 1. 2013)**

The non-thermal preservation methods could be easily combined with the traditional preservation methods to produce high quality food products. The new mild processing techniques used in this study include: osmotic dehydration, vacuum impregnation, pulse electric field and edible coatings.

Five raw materials with different tissue structure such as potato, carrot, Jerusalem artichoke, apple and strawberry were used for the osmotic treatment. The changes in tissue's density and porosity under different temperatures and concentrations of the osmotic solutions were evaluated. The effect of different parameters on the osmotic dehydration of the different plant tissues in terms of water loss and solid gain, were investigated. The water removal rate was in the range from 0.076 to 0.624  $\text{g}_{\text{water}}/\text{g}_{\text{SM}}\cdot\text{h}$ , while the solute uptake's rate was from 0.040 to 1.043  $\text{g}/\text{g}_{\text{SM}}\cdot\text{h}$ . The water diffusion coefficient was in the range of 0.8 to  $16\cdot 10^{-11}$   $\text{m}^2/\text{s}$ . Osmotic treatment deactivates the enzymes and it preserves well the aroma and the color, the mechanical and nutritive properties of the fruits and vegetables. The dried apples with well preserved optical and nutritive properties, having the common characteristics of crispy products were transformed in a new product, apple chips.

The effect of cryoprotectants on the freezing tolerance of strawberry tissues were evaluated by means of vacuum infusion and pulse electric field. The cryoprotectants used in this study were trehalose and antifreeze protein. They were vacuum infused in the strawberries' apoplast. The pulse electric field enabled direct impregnation of the plant cells with cryoprotective solutes. The tissue absorbed 9% and 14% of antifreeze protein and trehalose. When the combination of the both solutions was used the tissue absorbed 19%. The combined effect of both cryoprotectants significantly improved the freezing tolerance of the treated strawberries and maintained 90% viability of the plant cells after thawing.

The minimal processing of fruits was carried out with edible films. Edible coatings act as moisture and gas barriers and improve the quality and shelf-life of minimally processed fruits and vegetables. For that purpose 10 chitosan-based edible films were prepared. Chitosan films were modified by crosslinking, application of a beeswax coating on both interfaces of the film and preparation of composite films. All edible films were characterized in terms of their optical, mechanical, barrier, sorption and thermal properties. The incorporation of the beeswax into the edible films decreased the water vapour permeability value from  $4.15\cdot 10^{-11}$  to  $1.5\cdot 10^{-11}$   $\text{mol}\cdot\text{m}/\text{m}^2\cdot\text{s}\cdot\text{Pa}$ . The edible coatings as a preservation method were tested on strawberries. The coatings modified the respiration rates of the strawberries and slowed down their metabolism extending their shelf-life at 20°C. The three-layer coatings enabled the best protection of the overall quality of the strawberries, but the chitosan and composite coatings were more preferable for the consumers.

The emerging technologies in combination with the already existing methods besides providing microbial stability, fresh look and extended shelf life also gave better preservation of the sensorial and nutritional properties of the fruits and the vegetables.

*Keywords:* non-thermal processes, osmotic treatment, vacuum infusion, pulse electric field, edible films

Aneta Vasiljević-Šikaleska

**SHEAR INDUCED MICRO- AND MACROSTRUCTURAL CHANGES IN POLYMER BLENDS  
BASED ON POLY(VINYLCHLORIDE) WITH POLY(ALKYLMETHACRYLATES) AND THEIR  
EFFECT ON THE RHEOLOGICAL, MORPHOLOGICAL AND FINAL PROPERTIES  
(11. 2. 2013)**

The aim of this thesis was to study the structure development in polymer blends when they are forced to flow under specified stress fields and its influence on rheological and mechanical properties.

Binary polymer blends of polyvinylchloride (PVC) with polymethylmethacrylate (PMMA), polyethylmethacrylate (PEMA), polybutylmethacrylate (PBMA) and polypropylene (PP) with 90/10, 80/20, 70/30 and 30/70 were investigated. Also nanocomposite material based on PVC/PEMA 90/10 blend was prepared by incorporation of montmorillonite nanoclay.

Test samples were prepared in a molten state using a Brabender mixer and afterwards extruded at two different rotational speeds in a single screw extruder. The characterization of these materials was carried out by scanning electron microscopy (*SEM*), transmission electron microscopy (*TEM*), parallel plate oscillatory rheometry, dynamic mechanical thermal analysis (*DMTA*), dynamometric method, differential scanning calorimetry (*DSC*) and X-ray analysis (*XRD*).

*SEM* micrographs revealed homogenous and uniform dispersions without visible phase boundaries. The fracture surfaces of samples processed in a brabender changed from brittle and glassy for neat PVC to ductile with high extent of plastic deformation for the nanocomposite and blends. It was also observed that additional shearing of samples in the extruder caused structural reorganization and elongated domains oriented in the flow direction were detected.

Rheological measurements performed under steady shearing conditions and dynamic oscillatory shearing were used to evaluate the shear-dependent viscosity,  $\eta(\dot{\gamma})$ , the first normal stress coefficient,  $\psi_1(\dot{\gamma})$ , and the second normal stress coefficient,  $\psi_2(\dot{\gamma})$ . Analytical equations of these dependences ( $\eta(\dot{\gamma}), \psi_1(\dot{\gamma}), \psi_2(\dot{\gamma})$ ) known as material functions, with the exception of  $\psi_2(\dot{\gamma})$  which is zero, were found. Also expressions for the functions of the storage modulus,  $G'(\omega)$ , the loss modulus,  $G''(\omega)$ , and the complex viscosity  $\eta^*(\omega)$  were determined. According to these functions all samples are viscoelastic with a pseudoplastic character of the apparent viscosity and the first normal stress coefficient. Pseudoplasticity, assessed by the viscosity material function, is more expressed with samples obtained in the brabender. For example, the viscosity material function of PVC/PEMA 90/10 blend processed in the brabender is  $\eta_{ap} = 14\dot{\gamma}^{-0.6}$ , while the function for the same blend additionally sheared in the extruder is  $\eta_{ap} = 8(\dot{\gamma})^{-0.4}$ . This noticeable change in the values of the flow index, which is closely related to the material structure, arises from the flow induced microstructures caused by the different stress field occurring in the brabender and extruder. These results are consistent with those obtained from the morphological analysis.

Good correlation between the correspondent functions from steady shear flow measurements,  $\tau_{12}(\dot{\gamma})$  and  $N_1(\dot{\gamma})$ , and dynamic oscillatory measurements,  $G'(\omega)$ , and  $G''(\omega)$  is observed. This finding, being a property inherent in all viscoelastic materials, is very important because it can be used not only to test the suitability of different rheological constitutive equations and models but also may confirm a more convenient method to generate data from some polymer characteristics to others quite different or to prove the validity of data obtained from different measurements.

DMTA results show that viscoelastic properties of samples in a solid state are significantly influenced by the manner of mixing, i.e. how the components are brought together into a direct contact. Materials with noticeably higher storage modulus, from 20 to 40%, are obtained when the material is processed in the brabender instead of extruder.

Tensile properties were evaluated by the Young's modulus, tensile strength and elongation. As expected, due to the homogenous dispersion of spherically shaped domains when blends are processed in the brabender materials with enhanced elasticity are obtained. Contrary, during extrusion the elongation of domains generates more brittle material. These results entirely agree with those obtained from SEM, DMTA and parallel plate measurements.

It has to be emphasized that experimental results confirm the assumption that during the operation of shaping of polymer systems into semifinal or final products microstructural changes do happen that undoubtedly affect strongly material end properties. Among all, the rheological properties of the studied polymer blends were the most sensitive ones to the flow induced microstructural changes.

The addition of nanoparticles improved the rheological behavior of polymer blend in a molten and solid state, without worsening its processibility. This is of particular importance because indicates that the nanocomposite material can be processed using the same operations and processing conditions as those for neat polymer blends.

Based on a thorough analysis of the obtained experimental results, observations, analytical relations of the material functions and viscoelastic properties, others very specific findings and of course the current knowledge in this field, it is shown that the rheological behaviour, being the most sensitive to flow induced structural and morphological changes, remains the main concern in polymer processing especially when polymer blends are processed. It dictates the rate and quality of final products. Therefore the rheological viscous and elastic behaviour quantitatively determined in this investigation, with incorporated molecular parameters and engineering principles can be used as a fundamental input in the mathematical formulation of the process as whole. This approach along with advanced computational tools of modelling, simulation and optimization will enable achieving products with desired specific properties.

*Keywords:* Polymer blends, rheology, material functions, viscoelasticity, shear induced micro and macrostructural changes, mechanical properties

### 3

**Maja Dimitrovska**

#### **ANTHOCYANIN PROFILE OF THE MACEDONIAN RED WINES (10. 5. 2014)**

The anthocyanin composition of red grape varieties Vranec, Cabernet Sauvignon, Merlot and Pinot Noir grown in R. Macedonia was determined analyzing the skin extracts of the grape berries. The relative content of the identified anthocyanins was calculated and the obtained anthocyanin profiles were compared in terms of acylation and anthocyanidin distribution. The predominant anthocyanin in all varieties was malvidin-3-glucoside. Vranec variety was characterized by high proportions of coumaroyl derivatives and peonidins. The most pronounced difference among the studied grape cultivars was the relative amount of acetate derivatives. Different relationships among the anthocyanin groups of compounds were considered as parameters for differentiation of the cultivars. The relationship between coumarates and acetates in Vranec was significantly higher compared to Merlot and Cabernet. Moreover, the greatest difference of ratio between delphinidins and peonidins allows clear discrimination between the studied grape cultivars.

Evolution of the individual monomeric anthocyanins during vinification of grapes into wines was investigated using different oenological treatments for each of the studied grape varieties. The dynamics of the extraction was observed daily during the maceration, analyzing the anthocyanins in the must using HPLC. Additionally, the effect of the applied vinification protocols on the anthocyanin composition of the obtained wine at the end of maceration and wine aged 6 months was investigated and compared with the anthocyanin patterns of the original grape. The results confirmed that anthocyanin composition of the grapes changed during fermentation. Different

vinification protocols applied to a same raw material resulted in wines with similar anthocyanin patterns. However, the similarity decreased with the aging of the wines.

Wines of Vranec, Cabernet Sauvignon and Merlot varieties, from 1 to 3 years old, produced in different wineries in R. Macedonia were examined for their anthocyanin and chromatic profiles, total phenols, antioxidant potential and macro- and micro-elements. The established data were submitted to analysis of variance and principle component analysis in order to evaluate their potential for differentiation of wines according to variety and vintage. Vranec wines have shown distinctive characteristics, with the highest content of anthocyanins and values of colour intensity, % red and % dA, compared to the other two studied varieties. The content of petunidin-3-glucoside, peonidin-3-glucoside and anthocyanin acetates were established as possible markers for differentiation of Vranec wines from Cabernet Sauvignon and Merlot wines. However, none of the assayed parameters could be used for differentiation of Cabernet Sauvignon from Merlot wines. It was observed that successful classification of the wines by variety according to anthocyanins is limited by the wine age. The chromatic parameters allowed distinguishing of young from old Vranec wines. Additionally, it was found that elemental composition distinguished domestic wines from wines produced in other regions of the world.

*Keywords:* anthocyanins, anthocyanin ratios, grape skins, Vranec, Merlot, Cabernet Sauvignon, Pinot Noir, vinification, wine, colour characteristics, fingerprint, total phenols, antioxidant activity, macroelements, microelements, differentiation

**МАГИСТЕРСКИ ТРУДОВИ  
ОДБРАНЕТИ НА ТЕХНОЛОШКО-МЕТАЛУРШКИОТ ФАКУЛТЕТ  
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 Сарашов Митко Марјан, 9.10.2013  
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 Миленковиќ Љубиша Ружица, 15.10.2013

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Митевска Ацо Емилија, 25.1.2013  
 Андонова Борис Маја, 5.2.2013  
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 Ризов Никола Влатко, 22.2.2013  
 Димитриев Орданчо Никола, 10.5.2013  
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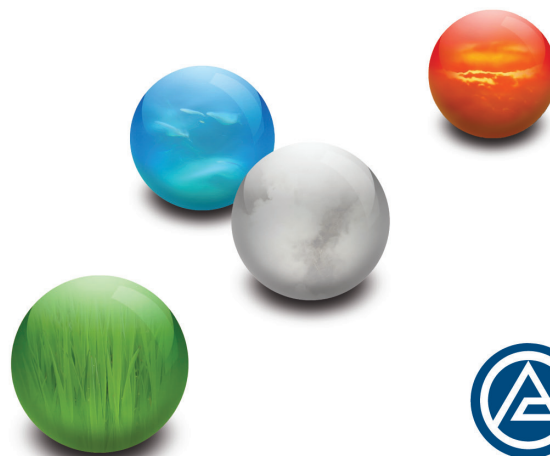


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## Agilent 7890B/5977 GCMS

Најновата серија на гасномасени хроматографи со кои се поместуваат лимитите на инструментите и се втемелуваат нови индустриски стандарди во оваа аналитичка дисциплина



## Agilent 6400 серија на LC-MS

Супериорна осетливост и неспоредлива ефикасност на сите модели од 6400 серијата на течно масени детектори со троен квадрупол од Agilent Technologies



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