AKTION ÖSTERREICH - TSCHECHISCHE REPUBLIK

AKTION ČESKÁ REPUBLIKA - RAKOUSKO

Final Report – Závěrečná zpráva – Abschlussbericht

Characterisation of complex systems for biological applications using advanced rheological and spectroscopical techniques (AKTION No. 73p17)

Participants of the project:

Scientists: Prof. Ing. Miloslav Pekar, CSc., Brno University of Technology (BUT)
Ass.-Prof. Milan Kracalik, Ph.D, Johannes Kepler University Linz (JKU)
Students: Ing. Romana Kratochvilova, BUT
Ing. Jiří Smilek, BUT
MA. Elke Bradt, JKU

The project AKTION No. 73p17 was realized in a period from 01. 07. 2015 to 31. 03. 2016 at the Brno University of Technology in Czech Republic and at Johannes Kepler University Linz in Austria. The main stress was put on hydrogel systems with potential applications in agricultural and environmental technologies because of still worsening state of fertile soils in Czech Republic and in Austria as well.

In the first part of project realization, samples of investigated complex systems were prepared. These samples are based on hydrogel structure and are suitable for many biological applications. They are commercially named as superabsorbent polymers and they can positively influence the water control of the soil and provide necessary humidity for plants. The preparation of these systems was aimed to incorporate some active material into the structure of hydrogel to create a system of controlled release of nutrients. The final system should provide not only the moistness but even the adequate intake of nutrients for crops. Pictures from the preparation of these samples can be found enclosed as Picture 1 – 4.

Eight of all prepared samples were assessed as suitable for further investigation. The selection was depended on their basic composition, running of preparation process, ability of swelling and apparent stability of final hydrogel. Swelling properties of prepared hydrogels were for other structural investigation crucial. Exact amount of prepared xerogel was immersed into excess of water and the ability of hydrogels' water uptake was compared. All samples exhibit very good swelling properties, what is displayed in Pictures 5 and 6. Fully swollen hydrogels were treated under further investigation.

The basic viscoelastic characterisation of fully swollen samples was determined using Rheometer Anton Paar Physica MCR 501 (Picture 7). The measurement was done at 30 ± 2 °C using a parallel plate system (PP25-SN6375, 25 mm diameter) at 1 mm gap. Viscoelastic measurements, oscillation – frequency sweep and strain sweep, were performed for each sample and the obtained values of moduli G´and G´´ were compared. Storage modulus G´ is proportional to the extent of the elastic component and loss modulus G´´ is rational to the extent of the viscous component of the system. The flexibility of materials is measured by the magnitude of tan δ (the ratio G´´/G´), where δ is a phase angle. If the value of tan δ is larger than 1, it means G´´ is higher than G´, the system behaves like a liquid. In contrary, if the value of tan δ is smaller than 1, it means G´´ is smaller than G´, the superabsorbent exhibits solid-like behaviour. Accordingly, the strength of the interaction or network structure is basically measured by the magnitude of tan δ. The smaller the tan δ is, the stronger 3D network is formed.

The time-lapse rheological experiment was running for a six weeks. All of investigated samples were placed into excess of water and their viscoelastic properties were measured in regular time intervals. No significant

change in viscoelastic properties of fully swollen samples was observed during this period of time. It means that our samples are mechanically stable and they will not degrade in several days after application. This theory should be confirmed by suitable soil experiment. Re-swelling of all studied samples was observed by rheological point of view. Samples were immersed into the excess of water. Fully swollen hydrogel was obtained after 24 hours of swelling. Surrounding water was replaced and swollen hydrogel was immersed into oven with condition of 50 °C and they were dried until reach of constant weight of xerogel structure. This xerogel was immersed to the excess of water again to swell for 24 hours. Swelled hydrogel were then treated under viscoelastic measurement. This cycle of measurement were repeated several times. The weakening of polymer crosslinked structure was objected with every single experiment cycle. But there is still fully functional hydrogel structure even after five cycles of re-swelling. Total collapse of hydrogel structure was not reached during undertaken experiments.

Viscoelastic properties of hydrogel samples were studies by novel microrheology method as well. Microrheology uses small amount of sample, typically in range of microliters which makes this method suitable in biological applications where small amount of sample is required. Classical microrheology method could not be used mainly because of its limitation caused by using of relatively large particles related with conventional microscope. Dynamic light scattering (DLS) by using ZetaSizer Nano ZS (see Picture 8) and fluorescence correlation spectroscopy (FCS) by using Time-resolved Fluorescence Microscope (Picture 9) were consider as a more suitable for studying of investigated samples because both spectroscopy methods enable to use nanometer sized particles. After comparison of both mentioned methods, the method FCS – microrheology was considered as a more suitable for objected samples. Several measurements were performed and through mean square displacement of particles were determined local deformation of samples. During this project valuable viscoelastic characterization of investigated samples was obtained. Due to shortening of project period our team was not able to run experiments under rheo - Raman device, which could clarify some unexplained assumptions of our investigated complex systems.

It is one of the most important finding, why the project AKTION between Brno University of Technology and Johannes Kepler University should continue. Moreover optimized microrheological methods could be used for the characterization of other hydrogel systems if the extension of the project will be approved.

Involved persons

Person	Activity	Home university	Visit
Romana Kratochvilova	Preparation of hydrogels samples, basic characterization, swelling experiments, realization of rheological tests (JKU), determination of viscoelastic properties of hydrogels, preparation of publication	BUT	4 weeks
Jiri Smilek	Realization of rheological tests (repeated swelling, time degradation), determination of viscoelastic properties, preparation of publication (JKU)	BUT	4 weeks
Miloslav Pekar	Giving lecture, preparation of further projects and subsequent cooperation (JKU)	BUT	5 days
Milan Kracalik	Giving lecture, preparation of further projects and subsequent cooperation (BUT)	JKU	8 days
Elke Bradt	Realization of microrheology experiments (dynamic light scattering, fluorescence correlation spectroscopy) (BUT)	JKU	8 days

Two lectures were given within the project framework. The first one was given by M. Pekar at the Johannes Kepler University in Linz (1.12.2016) named **Colloids Systems in Biological Applications**. The second one by M. Kracalik at the Brno University of Technology (15.12.2015) named **Advanced Rheology using**

Simultaneous Spectroscopical Methods aimed on special rheological techniques in polymer and biopolymer systems suitable for biological applications, including systems containing nanoparticles.

Accomplishment of goals

1) Manuscript of scientific publication based on this cooperation

Initial cooperation between Brno University of Technology and Johannes Kepler University resulted in one publication presented on Nanocon 2015 (Rheological Approach for Agricultural Hydrogels). This manuscript will be indexed in the scientific database (Web of knowledge) later this year. The manuscript presented on the conference is included as attachment.

2) Final thesis in the field of rheological and spectroscopical characterization

Based on the cooperation of participating universities, the final thesis aimed on microrheological characterization of hydrogels was proposed. The supervisor of the master thesis is Jiri Smilek and the final thesis should be defended at academic year 2017/2018.

3) Reports from each individual stay

From international internship of Jiri Smilek and Romana Kratochvilova were written the final reports. These reports are included into the final report as attachment.

Justification of changes in the financial expenses

1) Travel costs

Expected costs of Czech participants in Austria: 6600 CZK
Expected costs of Austrian participants in Czech Republic (accommodation): 21000 CZK

Real costs of Czech participants in Austria: 4803 CZK
Real costs of Austrian participants in Czech Republic (accommodation): 14000

2) Consumables

Expected costs: 17793 CZK

Real costs: 20200 CZK

Expected travel costs were reduced because of cheaper accommodation of Austrian participants in Czech Republic, also real travel costs of Czech participants in Austria were lower than we expected. Unfortunately, the real costs of consumables were higher than we expected because the prices of material in submitted project were calculated without VAT.

Applicant

Assist. Prof. Ing. Milan Kracalik, Ph.D,

Linz, Austria, 8th April, 2016

JKU JOHANNES KEPLER UNIVERSITÄT LINZ
Institute of Polymer Science

Institute of Polymer Science
Altenbergerstrasse 69 • 4040 Linz • Austria
Phone: +43 732 2468 7100 • www.jku.at/ips

signature

Joint applicant

Prof. Ing. Miloslav Pekar, Csc.

Brno, Czech Republic, 7th April, 2016

signature

Brno University of Technology
Faculty of Chemistry
Institute of Physical and Applied Chemistry
Purkyňova 118, 612 00 Brno
Czech Republic