Report on the outcomes of a Short-Term Scientific Mission[1]

Action number: CA17107

Grantee name: Jelena Tanasić

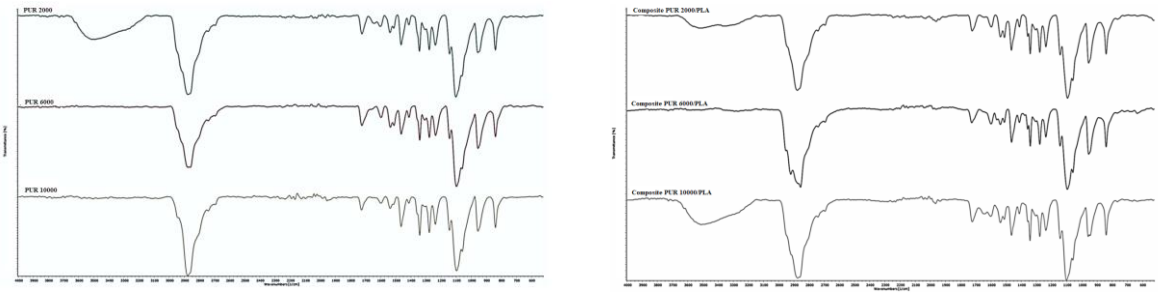
Details of the STSM

Title:Characterization of electrospun composite textile for biomedical application

Start and end date: 16/05/2022 to 27/05/2022

Description of the work carried out during the STSM

During my two-week STSM stay at Charles University, Faculty of Mathematics and Physics, Department of Macromolecular Physics in Prague, pure polyurethane hydrogels with three different polyethylene oxides like polyol components (PEO 2000, PEO 6000, and PEO 10000) and electrospun composite textiles based on polylactide and polyurethane hydrogel (PUR/PLA) for use in biomedicine were tested.
In the first week, samples were tested by IR spectroscopy and by Gel permeation chromatography (GPC) to confirm the presence or absence of monomers (especially isocyanates, which are undesirable in medical applications). Before characterization, samples were prepared by specific methods because of extraction from hydrogels structure. The results of IR spectroscopy show the absence of isocyanates in all samples (absence of peak for NCO group between 2200-2300 cm-1), both in pure hydrogels and in composites, whether the samples with PEO 2000, PEO 6000, or PEO 10000, figure 1.



a) b)

Figure 1. Results of FTIR method: a) pure hydrogels; b) electospun composite PUR/PLA

GPC analysis gives different results than FTIR method. The results showed the absence of monomers (in pure hydrogels and composites with PEO 2000), while the presence of isocyanates (Mw of isocyanate PMDI is 314 g/mol) was shown in samples with PEO 10000 (small amounts) and samples with PEO 6000

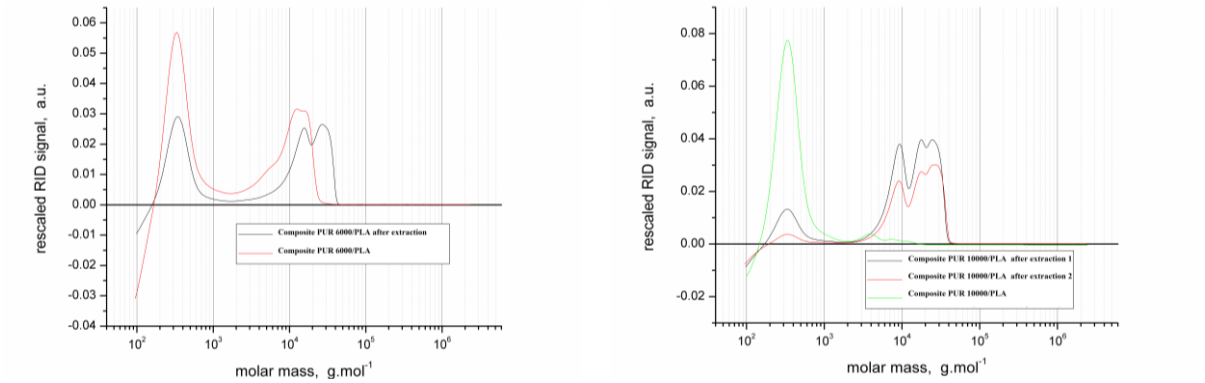
[1] This report is submitted by the grantee to the Action MC for approval and for claiming payment of the awarded grant. The Grant Awarding Coordinator coordinates the evaluation of this report on behalf of the Action MC and instructs the GH for payment of the Grant.

(large amounts). During the same week, the samples were put for purification from isocyanates, using hexane and tetrahydrofuran for extraction of isocyanate, figure 2, and then characterized at GPC.

a) b)

Figure 2. a) Process extraction of isocyanate from samples; b) samples after evaporating the solvent from samples

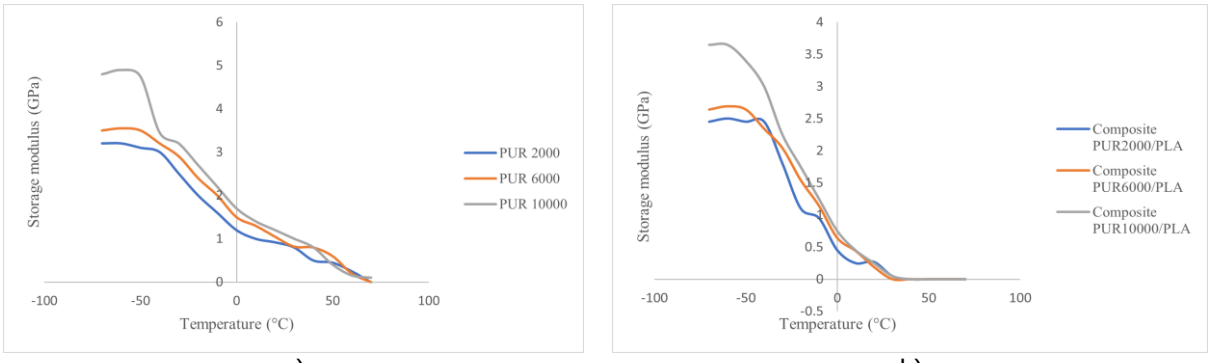
After these extractions, no isocyanate was present in the samples with PEO 10000, while isocyanate was still present in the samples with PEO 6000, figure 3.



a) b)

Figure 3. Results of GPC method for samples: a) with PEO 6000 and b) with PEO 10000

After both these methods, we concluded that both must be used to confirm the presence of monomers (GPC), and confirmation of polyurethane formation (IR method). Another method of characterization performed during the second week is dynamic mechanical analysis (DMA). The storage modulus (E ′) and loss (E ′ ′) were registered, for all composite samples and pure hydrogels. Figure 4 shows the results of the storage modulus. The highest values of storage modules in the entire range of tested temperatures were obtained for PUR 6000 and PUR 10000, whether they are pure hydrogels or composites with the same type of hydrogel. Analysis of results indicates that the softening process of the PEO segments begins at about 60 ◦C. This effect was observed for all samples and the softening point for composites was shifted to lower temperatures. Composite samples start softening at temperatures between 35 °C and 40 °C, this is a consequence of the way the film is made (electrospinning), but also due to the use of polylactides in composites, which is more thermolabile than polyurethane.



a) b)

Figure 4. Temperature dependence of storage modulus for samples: a) pure hydrogel and b) electrospun composite PUR/PLA

Description of the STSM main achievements and planned follow-up activities

The results obtained during this STSM at Charles University, Faculty of Mathematics and Physic, Department of Macromolecular Physics in Prague and the realized analyzes meet the planned goals of planned STSM. During my stay in Prague, significant results were obtained on the presence of monomers, more precisely isocyanates, in the samples, as this is one of the main problems that arose when testing samples of composites and pure hydrogels for use in medicine. This is an extremely important discovery because before this STSM and working with Professor Krakovsky, who is an expert in polyurethanes and working on GPC, I have not succeeded in any method I have used so far (IR spectroscopy, Raman spectroscopy, HPLC method, titration method) to confirm the presence of monomers in the sample (more precisely isocyanates) while the results of cytotoxicity showed that there were isocyanates or something else what kill all cells in the sample. The GPC method finally confirmed that the cells are killing the residual isocyanates and that research should go in that direction, to improve purifying of the samples. Also, during the stay, success was achieved in purifying part of the samples from residual isocyanates, which was confirmed by re-characterization at the GPC. Very significant results were also obtained at the DMA on the rheological properties of polyurethane hydrogel, as well as composites intended for use as a wound dressing. This method was necessary to confirm the possibility of using this electrospun composite textile for wound dressing. The realization of this STSM and the results obtained during the STSM are within the scientific realization of working group WG1: Smart textiles for health and medical applications. One of the goals for working group WG1 is the use of textile fibers in tissue engineering. The realization of this STSM allowed me to establish new contacts with Professor Krakovsky and his team at Charles University, Faculty of Mathematics and Physics, Department of Macromolecular Physics in Prague. During the stay, future cooperation was agreed upon on these samples, because together we will try to improve the method of purification of samples from residual monomers (isocyanates) as well as on some new projects and samples. Following this STSM, participation in the European Symposium for Thermal Analysis and Calorimetry is planned for August this year with the results of this STSM and some previous analyzes. It is also planned to publish the results in a journal with a high IF, as well as to submit applications for joint projects. This STSM and this collaboration are important for finishing my PhD thesis.