SHORT TERM SCIENTIFIC MISSION (STSM) SCIENTIFIC REPORT

This report is submitted for approval by the STSM applicant to the STSM coordinator

Action number: FP1405

STSM title: Development of hybrid cellulose nano-fibers/lignin particles films for active packaging applications

STSM start and end date: 28/01/2019 to 08/02/2019

Grantee name: Eva Pasquier

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| **PURPOSE OF THE STSM:** |
| This scientific mission aims to produce and characterize lignin particles to further incorporate them in cellulose nanofibers films and coatings. Cellulose nanofibrils (CNF) are well-known for their ability to make films with increased mechanical and optical properties. Furthemore, lignin has inherent anti-oxidant properties hence the final idea is to make anti-oxidant films for food packaging. This would make a fully bio-based and biodegradable material.  The group of Profesor O. Rojas has developed a method that allows to create perfectly round lignin particles directly in a dry state. The process prevents the aggregation of the particles which is usually the main problem with humid preparation method. Then the particles need to be characterized. Time permitting, cellulose nanofibers film containing lignin particles will be done. |

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| **DESCRIPTION OF WORK CARRIED OUT DURING THE STSM** |
| The aerosol flow reactor method was used to produce lignin particles of different sizes. For that purpose, 1 g of kraft lignin is dissolved in DMF, this solution is atomized under nitrogen. The gas flow brings the droplet in a oven heated at 153°C (boiling point of DMF) where they are air dried. The particles are then divided by size in a collector with 11 fractions. After 15 h reaction, the particles can be collected by size.  After preparation the particles were analyzed using different characterization methods. The DLS (Dynamic Light Scattering) was used to measure the particles size. SEM images of 3 different particles sizes were done to check the morphology of the powder. Zeta-potential was done to analyze the surface charge of these particles. To study the intercations between the lignin particles and the cellulose nanofibers, surface plasmon resonance (SPR) was conducted. The sensor was covered with one full layer of cellulose nanofibers TEMPO oxidized (the surface was further checked with AFM) and a suspension of lignin particles was injected. After flushing with water, it was possible to measure the adsorption of the lignin on the CNF.  Hybrid films with cellulose nanofibers were finally done using different mass ratios between lignin particles and cellulose nanofibers. Cross sections of these films were imaged with SEM to study the dispersion of the particles and the behavior of the film.  Visit of the labs and attendance to group meetings were also part of the mission as an integration in the team for further collaboration. |
| **DESCRIPTION OF THE MAIN RESULTS OBTAINED**    The lignin particles were prepared during one night but the experiment stopped during the nightn therefore the amount collected was lower than expected.  SEM images were done on fraction 2, 5 and 8 (1 being the smallest particles and 11 the biggest), we obtained really round and smooth particles. But the images show quite polydispersed particles in size even if the average increases with the increasing fraction (Figure 1). The partciles size measured on the SEM pictures are 140 ± 44 nm for fraction 2, 256 ± 171 nm for fraction 5 and 628 ± 577 nm for fraction 8. From the standart deviations and the size distribution, we could tell that the big particles are well collected in the first stages of the collector but the small particles are present at every stages.  F:\SEM\with info bar\A511.tif  Figure 1: SEM image of the fraction 5 of the lignin particles produced by the aerosol flow reactor  The SPR result show that the lignin particles adsorb on the CNF surface. After injection of the particles, we observe a high adsorption on the fibers; it increases slowly while continuously injecting the particles. After rinsing, most of the particles desorb but a small amount stays on the surface. The quantity of adsorbed particles is 5.6 µg/cm².  The cross-sections of the films demonstrate very interesting results, in fact depending on the lignin concentration in the film, the structure changes. With low concentration (9 wt%) of lignin the film keeps the structure of a CNF TEMPO film but with higher concentrations of lignin particles (33 wt%) the film has a new structure.  To go further with this work, the anti-oxidant properties of the films will be analysed using the DPPH test. It calculates the anti-oxdative power of a film using the molecule di(phenyl)-(2,4,6-trinitrophenyl)iminoazanium as radical scavenger. The barrier properties against oxygen can also be studied. |
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| **FUTURE COLLABORATIONS**  This PhD work is co-funded between LGP2 and Aalto University so the collaboration will continue. Monthly meetings take place between the two partners to present the results and discuss about the on-going work. Other visit will be carried along the PhD to enhance the foster the collaboration. |
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