STSM report COST FP1404

"Synthesis of nanocomposite materials with improved fire retardant/resistant properties"

Host: Prof. Holger Militz, Institute of Wood Biology and Wood Technology of the Georg-August-University of Göttingen

Period: from 05th of July to 15th of August 2016

Place: Göttingen, Germany

By: Dr. Carmen–Mihaela Popescu, 2015. 09. 10

Purpose of the visit to Institute of Wood Biology and Wood Technology of the Georg-August-University of Göttingen was to perform (together with the research group from there) a series of synthesis of environmentally friendly materials with improved performances for wood, with a special target to reduce its flammable ability. At the same time, this opportunity has been used to start the collaboration with Prof. Holger Militz and his group.

Short summary of performed STSM

In order to meet today's increasingly stringent demands for fire safety as well as product durability, and to limit the losses, different fire retardants and intumescent coatings are used. The growing awareness of environmental issues and consumer safety of fire retardant products increases the request for new, innovative materials for the coating industry more eco-friendly and compatible to the applied substrate.

In this context, the performed STSM proposes to develop materials with improved performances for wood, with a special target to reduce its flammable ability. Therefore, during this period, a series of different polymeric solutions based on an acrylic polymer / copolymer, diammonium phosphate, pentaerythritol, melamine, benthonite, nano crystalline cellulose, montmorillonite K10, and a nitrogen phosphorous compound (NP) were synthesized. After synthesis, the best obtained solutions were used to impregnate spruce sapwood samples. In order to evaluate the successful impregnation process in the wood samples, scanning electron microscopy and infrared spectroscopy were performed, and in order to test their efficiency was used thermogravimetry.

The project of the Short Term Scientific Missions (Exchange visits) in the COST Action FP1404 framework was also used to start the collaboration with Prof. Holger Militz at the Institute of Wood Biology and Wood Technology of the Georg-August-University of Göttingen.

Description of the main results obtained

During the mentioned STSM a series of polymer composites were synthesized, by varying both the polymer matrix and the composition of the listed materials (see Table 1).

Table 1. Prepared polymeric solutions and impregnated wood

Name	I	II	III	IV	V	VI	VII	VIII	IX	Observation	Impreg. wood
Sa		---								c=10%	yes
Sb	---									c=10%	yes
III			---							c=20%	yes
IV				---						c=1%	
V					---					c=10%	
VI						---				c=0,5%	
VII										c=1%	
VIII								---		c=0,5%	
IX									---	c=5%	
Sc			---	---	---					57.1% : 14.3% : 28.6%, c=5%	yes
S1		---	---							71.5% : 28.5%, c=11.6%	yes
S2		---	---		---					60.9% : 26.3% : 12.8%, c=11.5%	
S3	---		---							71.4% : 28.6%, c=11.6% separation	
S4		---	---	---	---					61% : 24.4% : 12.2% : 2.4%, c=9.1%	yes
S5		---	---	---	---		---			60.2% : 24.1% : 12.1% : 2.4% : 1.2%, c=8.3%	
S6		---	---	---	---	---				58.1% : 23.2% : 11.6% : 5.8% : 1.2%, c=6.2%	
S7		---							---	66.6% : 33.4%, c=13.6% for wood impregnation diluted 50:50 with water	yes
S8		---				---			---	65.8% : 32.9% : 1.3%, c=10.2%	
S9	---								---	66.6% : 33.4%, c=13.6%	yes
S10	---					---			---	65.8% : 32.9% : 1.3 %, c=10.2% Phase separation	
S11	/VII		---							used CNC in the polymerisation process, after added DAPH precipitation oft he polymer	
S12		---	---	---	---		---			58.1% : 23.2% : 11.6% : 5.8% : 1.2%, c=6.6%	yes
S13	---		---	---	---		---			59.1% : 23.7% : 2.4% : 11.8% :	

										3%, c=6.1%	
S14	---		---	---	---	---				59.1% : 23.7% : 2.4% : 11.8% : 3%, c=6.1% Phase separation	
S15	---		---	---	---					58.8% : 23.5% : 5.9% : 11.8%, c=7.5% Oil phase separation – polymer at the surface !?	
S16	---		---	---						66.7% : 26.6% : 6.7%, c=7.2% Oil phase separation but not that pronounced as in the previous case	
S17		---	---	---	---					55.6% : 22.2% : 11.1%, c=5.5%	yes
S18		---	---				---	---		70.4% : 28.2% : 1.4%, c=8.9%	yes
S19	---		---					---		70.4% : 28.2% : 1.4% c=8.9% Oil phase separation at the surface of the solution	
S20		---	---	---				---		65.8% : 26.3% : 6.6% : 1.3%, c=5.8%	yes
S21		---	---	---				---		35.6% : 54.8% : 7.5% : 2.1%, c=5.6%	yes
S22	/VII		---	---						used CNC in the polymerisation process, after added DAPH and MEL precipitation of the polymer	

I - poly buthyl methacrylate II - methacrylic acid 23
III - diamonnium phosphate IV - melamine
V - pentaerythritol VI - benthonite
VII - nano crystalline cellulose VIII - montmorillonite K10
IX - nitrogen phosphorous compound (NP)

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In order to test the structure and their efficiency, the obtained composite materials were prepared in film form (by solvent casting method at room temperature) and also some of them were used for the impregnation of the spruce sapwood samples.

Both, the polymeric films and the impregnated wood samples were analyzed through the thermogravimetric ananlysis and infrared spectroscopy.

The presence of the polymeric film at the surface of the wood was firstly evidenced by the scanning electron microscopy analysis. In Figure 1 is given one example of the treated wood with Sc and S9 solutions.

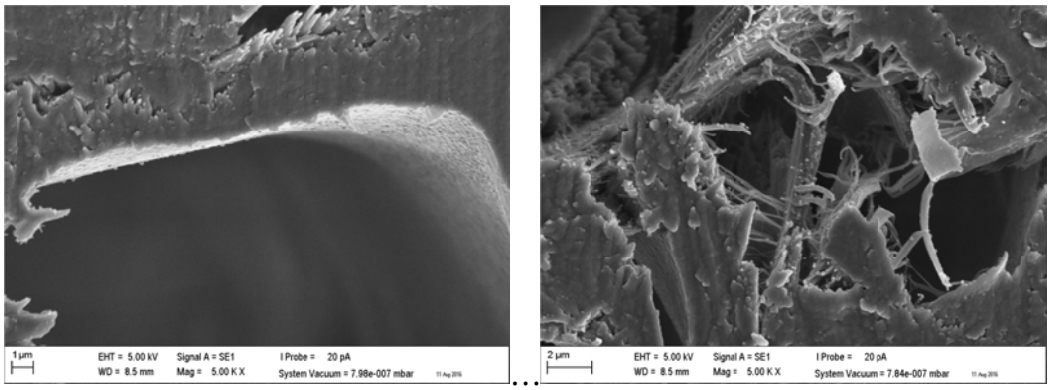

Figure 1. Scanning electron microscopy images of treated wood with the Sc (a) and S9 (b) solutions

Further, infrared spectroscopy was used in order to indicate the presence of the polymeric film at the surface of the wood samples. In Figure 2, the differences in the spectra of the untreated (reference) and treated wood samples can be observed, indicating the presence of the component materials present in the impregnating material.

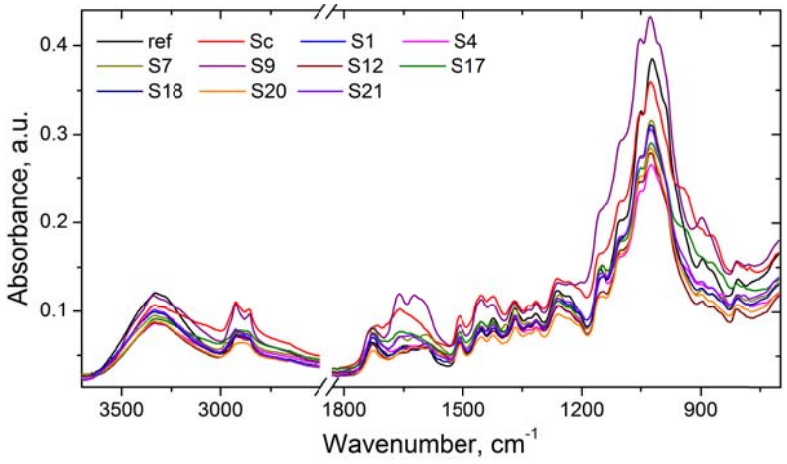

Figure 2. FT-IR ATR spectra of reference wood and treated wood with different polymeric solutions

The preliminary results of the TG curves (Figure 3) indicate an increase of the residual mass at the end of the stage for the wood samples treated with the previous listed chemicals, from about 10% (residual mass) for the untreated wood sample up to about 30 % for the wood treated with S17. At the same time, the temperatures for the maximum rate loss for the decomposition process were changed according to the coating used.

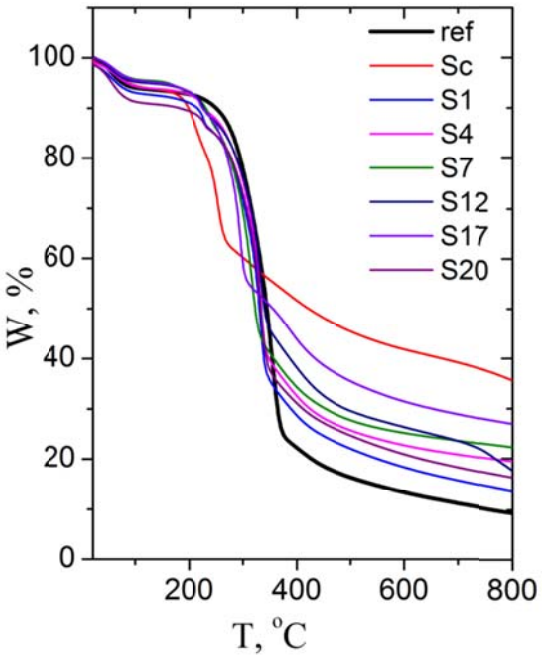

Figure 3. TG curves for the spruce wood impregnated with different solutions

Preliminary results indicate that the obtained solutions increase the protection of wood against fire, but of course further analysis and other methods needs to be used in order to prove their efficiency.

Future collaboration with host institution

My collaboration with the host institution, especially with Prof. Holger Militz, will continue also after this STSM trough future investigations concerning the synthesized materials during the COST FP1404 Action and also after the Action will end.

In the near future, we are also looking for new application project calls (national or H2020) in order to be able to continue the started investigations.

Projected publications / articles resulting or to results from the STSM

We will prepare a presentation for a future COST meeting and also, if satisfactory results a co-authored manuscript might be written and sent for publication in a peer reviewed journal.

Other comments

During the period of my stay apart of the experiments, I had discussions on the STSM topic with colleagues from the Institute of Wood Biology and Wood Technology. The period

of STSM was significant and creative for me, allowing me to exchange knowledge and research experiences.

I would like to thank to COST office and COST FP1404 committee to open this opportunity to collaborate with Prof. Holger Militz from the Institute of Wood Biology and Wood Technology of the Georg-August-University of Göttingen. We intend to continue collaboration both during COST FP1404 period and also by other collaborations.

I would like also to thank Prof. Holger Militz and Michael Altgen for their support and help during the STSM.