



SCIENTIFIC REPORT
Workshop NANO-NOCMAT # 1
10-12 Sep 2018 Pirassununga, Brazil



Nanotechnology Applied to Construction Materials

Coordination: Holmer Savastano Jr (University of Sao Paulo, Pirassununga Brazil)

From September 10th – 12th the Workshop NANO-NOCMAT # 1 Nanotechnology Applied to Construction Materials was held in Pirassununga, São Paulo State, Brazil, in the University of São Paulo (USP), Campus Fernando Costa. The complete workshop schedule is presented in **Annex 1**.

The event is part of the INCOBRA – Increasing International STI Cooperation between Brazil and the European Union. During the workshop, different activities took place: lectures and discussions, laboratory practices, presentation of the research groups linked to the project, as well as meetings with the members to propose a project of cooperation between Brazilian and European institutions (Horizon 2020 – H2020). The list of the original members of the NANO-NOCMAT project is described in **Annex 2**.

The profile of the public that participated in the event was mainly integrated of undergraduate and graduate students from Brazilian universities. The complete list of participants is described in **Annex 3**.

The presentations of the event, photos and information are available at the links:

- ✓ https://www.youtube.com/channel/UCKGsqCSGPWAO14Y7vFU_rwA,
- ✓ <http://sites.usp.br/biosmat/en/english-incobra-project/>
- ✓ <https://www.facebook.com/incobra.nanonocmat.5>

The following activities developed in each day of the event, the presentations of the members and the topics covered are described in the following sections of this report.

First Day 10th Sep

- Opening ceremony – carried out by the Deputy Dean of FZEA USP Pirassununga, Prof. Carlos Ambrosio.
- Course 1 – Biomimetics. Professor Anandi R. Sanadi, University of Copenhagen, Denmark. Concepts and results of the research focused on biomimetics applied to design of composite materials for civil construction and biomaterials.
- Course 2 – New techniques for the characterization and evaluation of mineral additions in fiber-cement composites. Professor Ernesto Villar Cociña, Central University “Marta Abreu” in Las Villas, Cuba. This participation was possible thanks to the FAPESP support (Visiting Foreign Researcher Grant).
- Course 3 – Nanocellulose. Dr. Gilberto Siqueira, Swiss Federal Laboratories for Materials Science and Technology (EMPA), Switzerland. Concepts and methodologies for the production and application of nanofibers and cellulose nanocrystals in 3D structures for, e.g., civil construction purposes. In addition, Dr. Siqueira coordinated a laboratory practice where the pre-treatment of cellulose with TEMPO reagent was demonstrated. This pre-treatment aimed to optimize the production of nanofibers via grinding process. The practice was in the Rural Construction Laboratory (CONSTRAMBI Lab), FZEA USP and the activities script of the laboratory practice is presented in **Annex 4**.

Second Day 11th Sep

- Presentation of the member institutions of NANO-NOCMAT INCOBRA Network, researchers and research lines focused on non-conventional materials applied to civil construction. The universities presented were:
 - ✓ Universidad de Extremadura, Spain (Dr. César Medina Martínez) and partnering unity;

- ✓ Instituto Español del Cemento y sus Aplicaciones, Spain (Dr. Miguel Ángel Sanjuán);
 - ✓ Ege University, Turkey (Dr. Candas Adiguzel Zengin);
 - ✓ Marmara University, Turkey (Prof. Meral Birbir and Dr. Pinar Caglayan);
 - ✓ Université des Antilles, Guadeloupe, France (Prof. Marie-Ange Arsène);
 - ✓ Swiss Federal Laboratories for Materials Science and Technology (EMPA); Switzerland (Dr. Gilberto Siqueira);
 - ✓ Leather and Footwear Research Institute (ICPI), Romania (Dr. Viorica Deselnicu);
 - ✓ University of Copenhagen; Denmark (Prof. Anand Ramesh Sanadi);
 - ✓ Politehnica University of Bucharest, Romania (Dr. Dana Corina Deselnicu);
 - ✓ Universidade Federal de Lavras, Brazil (Dr. Gustavo Henrique Denzin Tonoli);
 - ✓ Universidade Estadual Paulista Júlio de Mesquita Filho, Ilha Solteira campus, Brazil (Prof. Jorge Luís Akasaki);
 - ✓ Instituto Nacional de Pesquisas da Amazônia (INPA), Brazil (Dr. Ruy Alexandre de Sá Ribeiro and Dr. Marilene Gomes de Sá Ribeiro).
- Continuation of the mini-courses by the invited guests:
 - ✓ Course 4 – Leather wastes applied for civil construction and circular economy. Dr. Viorica Deselnicu, Leather and Footwear Research Institute, Bucharest, Romania. Examples of different types of leather wastes and alternative applications in materials for civil construction purposes.

- ✓ Course 5 – Life cycle assessment (LCA) for evaluation and analysis of environmental performance of new materials and use of LCA programs. Dr. Dana Corina Deselnicu, Politehnica University of Bucharest, Romania. Examples of available life cycle assessment for the production chain of the leather used in the production of footwear.

Third Day 12th Sep

- Presentation by TNS – Antimicrobial Solutions. Eng. Vendelino Oenning Neto.
- Presentation by the INCOBRA Manager Chiara Davalli (EBN, Belgium). Important topics of INCOBRA, e.g., networks involved in this cooperation, the mentoring and co-mentoring scheme and consultancy services offered.
- Presentation about the Project Horizon 2020 (H2020). Laura Rivero Garcia (CSIC). The presentation covered topics related to how to elaborate and submit a proposal for the project H2020.
- Presentation of the INCOBRA Co-mentor Luciana Lenhari (UNICAMP). Co-funding scheme, coordination and alignment of research and innovation funding and instructions for the H2020 proposal.
- Presentation by the International Office of FZEA-USP (CRInt). Guidelines for international academic agreements and memorandum of understanding (MoU).
- Presentation by Prof. Holmer Savastano Junior. H2020 Project and potential calls for H2020 proposal.
- Discussion of the project proposal and coordination with all network members.
- Technical visits and wrap-up activities in the CONSTRAMBI Lab. The participants went for a visit to the Rural Construction Lab for following the ongoing project activities and available infrastructure. They were also offered a tour in the Pirassununga Campus facilities, with special attention to the

Department of Biosystems Engineering and Department of Food Engineering,
FZEA USP.

Workshop NANO-NOCMAT in numbers

Figure 1 presents the statistics of the participants, while Figure 2 presents the distribution of the institutions that participated in the 1st Workshop NANO-NOCMAT. The NANO-NOCMAT Network covered a large number of graduation and undergraduation students, with a total number of 50 attendants, including the foreign visitor. Besides, during the meetings different institutions as São Paulo University (USP), Federal University of Lavras (UFLA), São Paulo State University (UNESP), São Carlos Federal University (UFSCar), Brazilian Institute for Amazonian Research (INPA) and others were attending.

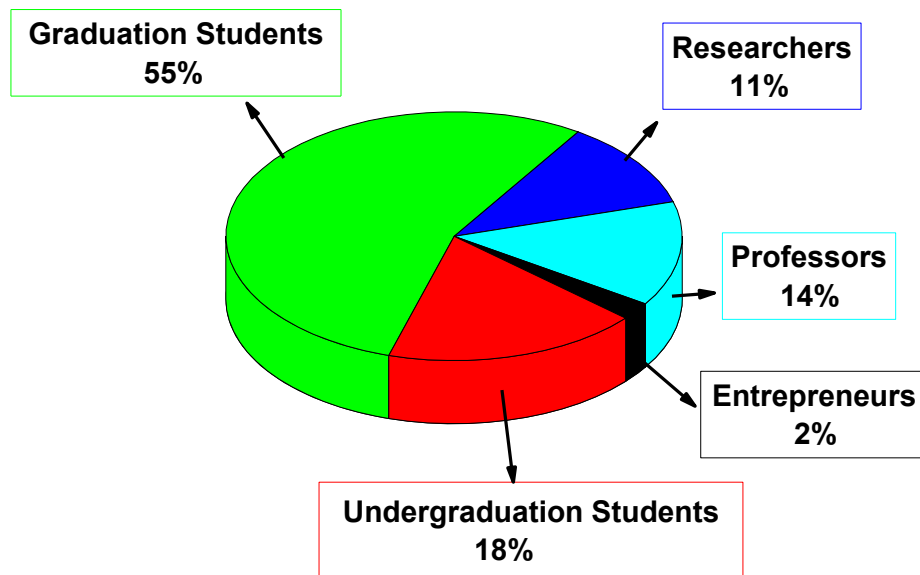


Figure 1. Distribution of the participants in the 1st Workshop NANO-NOCMAT

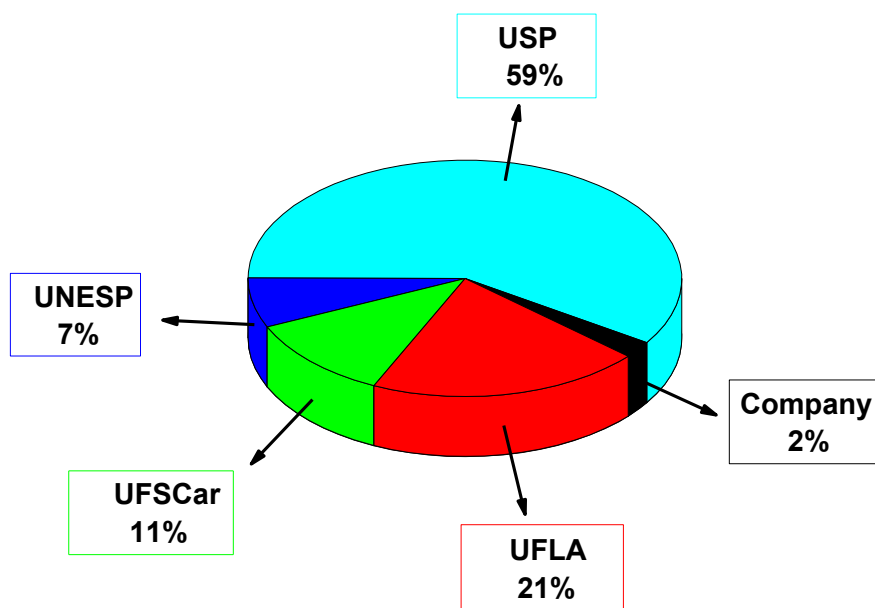


Figure 2. Distributions of Brazilian Institutions participating in the 1st Workshop NANO-NOCMAT

NANO-NOCMAT Network activities

The NANO-NOCMAT Network activities were defined for the near future in a timetable summarized in Figure 3. This sequence of activities was defined as the result of the 10-12 Sep 2018 meetings.

The participation in the Community Event and INCOBRA Training on Proposal Writing, Vienna, Austria, Sep 26th, 2018, will allow improving the preparation of the proposals and contributions from mentorship and supporting institutions.

Another concomitant event was the course on Bamboo in the Constructed Environment, offered from Sep 12-13, 2108. This course was important to attract graduate students interested in related topics of nonconventional materials and techniques. The budget for paying the participation cost of Dr. Bhavna Sharma (University of Bath, UK) and Prof. Khosrow Ghavami (PUC Rio, Brazil) was facilitated by FDTE, CAPES and Graduate Provost USP, Brazil.

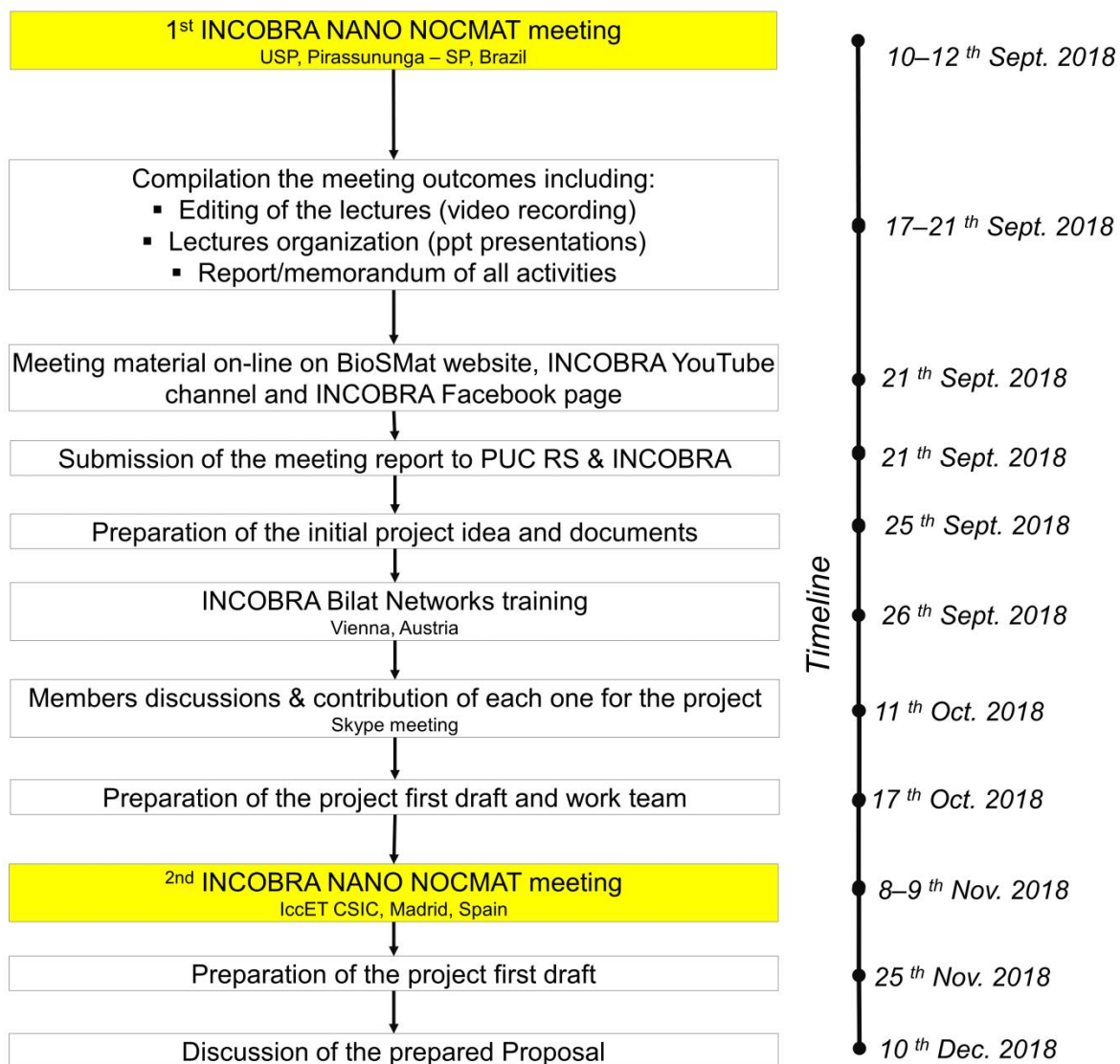


Figure 3. Timetable chart for the NANO-NOCMAT Network.

NANO-NOCMAT on social media and photos

Figures 4 to 7 illustrate the activities carried out during the 1st meeting of the NANO-NOCMAT Network from Sep 10-12 and other related activities in Pirassununga, SP, Brazil. Other pictures could be found in the social account: <https://www.facebook.com/incobra.nanonocmat.5>



Figure 4. Participants of the 1st NANO-NOCMAT Network meetings.



Figure 5. Practical activities and discussions during the parallel courses



Figure 6. Spatial bamboo structure constructed by the participants of the intensive course “Bamboo in the constructed environment”.

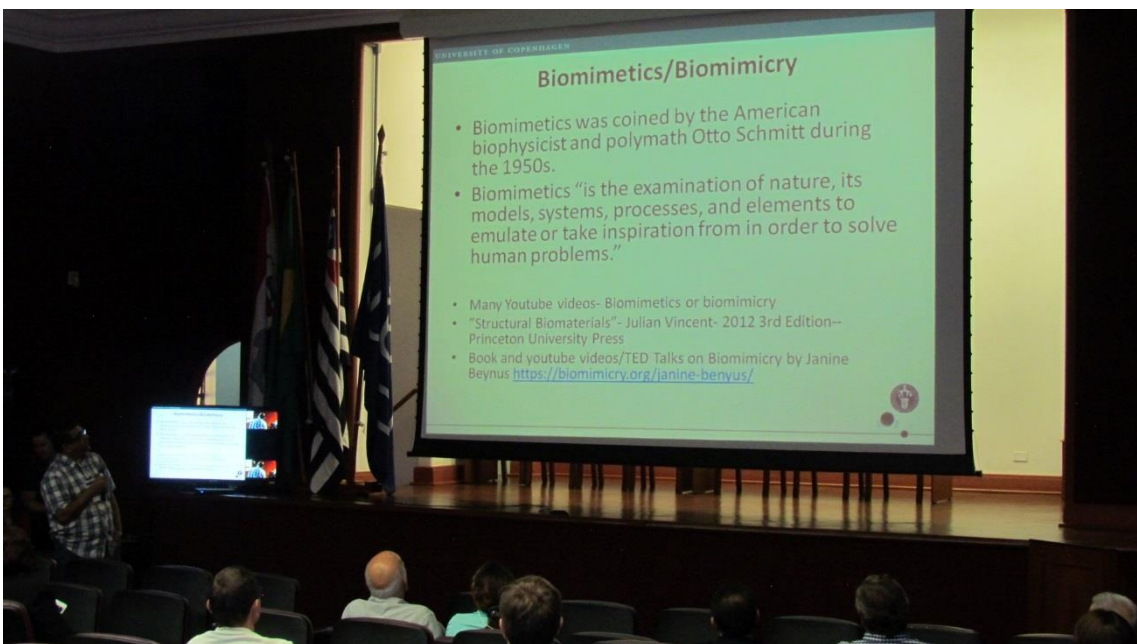


Figure 7. Presentation by Prof. Anand Sanadi (UCopenhagen) during the NANO-NOCMAT mini-course activities.

Document Repository

The presentations of the event are available in the YouTube NANO-NOCMAT channel: https://www.youtube.com/channel/UCKGsqCSGPWAO14Y7vFU_rwA

Table 1 presents the links available of the videos as well as the materials (pdf with slides of the presentations) of each lecture.

Table 1. Document Repository of the 1st NANO-NOCMAT Workshop.

1st Day (10/09/2018) – Short Courses		
Anand R. Sanadi, University of Copenhagen, Denmark. Course 1: Biomimetics – General concepts with examples	Video and PDF Presentation	Not available *
Ernesto Villar Cociña, Central University "Marta Abreu" of las Villas, Cuba. Course 2: Implementation of new techniques for the characterization of mineral additions in fiber-cement".	Video Presentation	https://youtu.be/fbOqh5hYSx0
Gilberto Siqueira, EMPA, Switzerland. Course 3: A biomass valorization for the production of nanocelluloses (nanofibers and nanocrystals)	Video and PDF Presentation	Not available
2nd Day (11/09/2018) – Morning – Presentations of INCOBRA members		
Cesar Medina, Universidad de Extremadura, Spain	Video Presentation PDF Presentation	https://youtu.be/zM7a3tb2SEc http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Cesar-Medina-presentation-1.pdf
Miguel Angel Sanjuan, Instituto Español del Cemento y sus Aplicaciones – IECA, Spain	Video Presentation PDF Presentation	https://youtu.be/49XOT8SrARw http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Presentacion-del-IECA-2018-MASanjuan.pdf
Candas Adiguzel, EGE University, Turkey	Video Presentation	https://youtu.be/HtDRfE-wiqQ
Meral Birbir and Pinar Caglayan, Marmara University, Turkey	Video Presentation	https://youtu.be/u9Ox0v3ce-U
Marie-Ange Arsène, Université des Antilles, Guadeloupe	Video Presentation	https://youtu.be/bqLE_fueJ5E
Jorge Akasaki, São Paulo State University – UNESP, Brazil	Video Presentation	https://youtu.be/6u6eUur_mr4
Gilberto Siqueira, EMPA, Switzerland	Video and PDF Presentation	Not available
Viorica Deselnicu, Leather and Footwear Research Institute, Bucharest, Romania	Video Presentation	https://youtu.be/wAF4PJnAq58
Gustavo H. D. Tonoli, Federal University of Lavras – UFLA, Brazil	Video Presentation	https://youtu.be/zOLRKeaSafo
Ruy Sá Ribeiro and Marilene Sá Ribeiro, National Institute of Research of the Amazon, INPA, Brazil	Video Presentation PDF Presentation	https://youtu.be/LCI9Vn2x2Qs http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Ruy-de-S%C3%A1-Ribeiro-GPBF-NanoNOCMAT-.pdf
Anand Sanadi, University of Copenhagen, Denmark	Video and PDF Presentation	Not available
Dana Corina Deselnicu, Polytechnic University of Bucharest, Romania	Video Presentation	https://youtu.be/Jf1MKc9HJLE

2nd Day (11/09/2018) – Afternoon – Short Courses		
Viorica Deselnicu, Leather and Footwear Research Institute, Bucharest, Romania. Course 4: Utilization of leather wastes for civil construction in the perspective of the circular economy.	Video Presentation	https://youtu.be/TiBLjltfOuM
Dana Corina Deselnicu, Polytechnic University of Bucharest, Romania. Course 5: LCA as an innovative tool for evaluating the environmental performance of new materials.	Video Presentation	https://youtu.be/UNe4K11lrJ8
3rd Day (12/09/2018) – Presentations and Meetings		
Vendelino Oenning Neto (TNS), Brazil	PDF Presentation	http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Pitch-TNS-1.pdf
Chiara Cavalli, EBN, Belgium	Video Presentation	https://youtu.be/T1Fti92Voc8
	PDF Presentation	http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/INCOBRA_NANO-NOCMAT-meeting_12-sept-2018_Chiara-Davalli.pdf
Laura Rivero Garcia, CSIC, Spain	Video Presentation	https://youtu.be/cn5a3uw8ILs
	PDF Presentation	http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/H2020-Laura-Rivero-12-09-2018.pdf
Luciana Lenhari, UNICAMP, Brazil	Video Presentation	https://youtu.be/qwOaIVDMM1k
	PDF Presentation	http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Nano-event_INCOBRA_Unicamp_Luciana.pdf
Keithy Domingos, USP, Brazil	Video Presentation	https://youtu.be/tpSgFjGNDY4
Holmer Savastano Junior, USP, Brazil	Video Presentation	https://youtu.be/NiwaJbtxcbY
	PDF Presentation	http://sites.usp.br/biosmat/wp-content/uploads/sites/98/2018/09/Holmer-Horizon-2020.pdf

(*) Video and PDF presentations will be available in the NANO-NOCMAT website when authorized by the respective presenters.

Final Remarks

The 1st Meeting of the NANO-NOCMAT Network was intense in activities and achievements. Several courses attracted students, researchers, academics and professionals with expertise in related topics with Nanotechnology and interest in actively taking part of the scientific proposals under construction. The Network members found an enjoyable and favorable environment to know each other, foreseeing synergic work in the near future and committing themselves with the calls for proposals. This was also important to bring managing and mentoring of INCOBRA Program closer of our Network with great insight to improve our ongoing work.

The Agreement with PUC RS was finally signed, thanks to the assistance of the group led by Prof. Marcírio Chaves, and the budget was released just in time for the

payment of air-tickets for the network members coming from Europe and other related costs.

The organizers are also asking to the participants to answer a Survey about their satisfaction and also for suggestions to the next meeting. We hope to have this Survey available in the near future until October 15th.

The proposed activities also were organized for the near future, as the time is considered very short for the great number of activities ahead on preparation of proposals for H2020 and ERAMIN2 calls. We are grateful and acknowledge all the support and participation of Network members and managers so far.

Pirassununga, SP, Brazil, 21st of September, 2018.



Holmer Savastano Junior

Annex 1- Event Full Program

INCOBRA MEETING - Workshop NANO-NOCMAT #1

1st Day 10th Sep		
Time	Activity/Session	Presenter
08:50 – 90:00	Opening Ceremony	Prof. Carlos Ambrosio, USP, Brazil
09:00 – 09:50	Biomimetics- A General Talk with Examples - Part A	Prof. Anand R. Sanadi, University of Copenhagen, Denmark
09:50 – 10:40	Biomimetics- A General Talk with Examples - Part B	
10:40 – 11:00	Discussion	
11:00 – 11:45	Implementation of new techniques for the characterization and evaluation of mineral additions in fiber-cement	Prof. Ernesto Villar Cociña, Central University “Marta Abreu” of Las Villas, Cuba
11:45 – 12:45	Biomass valorization for the production of nanocelluloses (nanofibers and nanocrystals) – Part A	Dr. Gilberto Siqueira, EMPA, Switzerland
12:30 – 14:00	Break: Lunch	
14:15 – 17:30	Production and applications of nanocellulose-based composites and practical activities – Part B (Offered in the CONSTRAMBI Laboratory)	Dr. Gilberto Siqueira, EMPA, Switzerland
2nd Day 11th Sep		
Time	Activity/Session	Presenter
09:00 – 12:30	Workshop	Presentations of the NANO-NOCMAT INCOBRA members
12:30 – 14:00	Break: Lunch	
14:00 – 15:00	Utilization of leather wastes for civil construction in the perspective of the circular economy – Part A	Dr. Viorica Deselnicu, Leather and Footwear Research Institute, Bucharest, Romania
15:00 – 15:30	Leather wastes as possible raw material for construction materials – Part B	
15:30 – 16:00	Discussion and practical activities	
16:00 – 17:00	LCA as an innovative tool for evaluating the environmental performance of new materials – Part A	Dr. Dana Corina Deselnicu, Politehnica University of Bucharest - UPB, Romania
17:00 – 17:30	Available LCA software programs and uses – Part B	
17:30 – 18:00	Discussion and practical activities	
3rd Day 12th Sep (work meeting)		
Time	Topic	Presenter
09:00 – 10:00	Managing	Chiara Davalli (EBN)
	H2020 mentor	Laura Rivero Garcia (CSIC)
	Co-mentor	Luciana Lenhari (UNICAMP)
10:00 – 10:30	Outline of the project proposal	Holmer Savastano (USP)
10:30 – 12:30	Discussion of the project proposal	All network members
12:30 – 14:00	Break: Lunch	
14:00 – 18:00	Technical visits (CONSTRAMBI Lab, Dept. Biosystems Eng. and Dept. Food Eng.) and wrap-up activities	All network members

Annex 2- NANO-NOCNMAT INCOBRA Network

Network Members and respective institutions

1. **Cesar Medina** (Universidad de Extremadura) and Partenering Unit;
2. **Miguel Angel Sanjuan** (IECA - Instituto Español del Cemento y sus Aplicaciones);
3. **Candas Adiguzel** (EGE University);
4. **Meral Birbir** and **Pinar Caglayan** (Marmara University);
5. **Marie-Ange Arsène** (Université des Antilles);
6. **Jorge Akasaki** (UNESP - University of the State of São Paulo);
7. **Gilberto Siqueira** (EMPA - Swiss Federal Laboratories for Materials Science and Technology);
8. **Viorica Deselnicu** (ICPI - Leather And Footwear Research Institute);
9. **Gustavo Tonoli** (UFLA - Federal University of Lavras);
10. **Ruy Sá Ribeiro** and **Marilene Sá Ribeiro** (INPA - National Institute of Research of the Amazon);
11. **Anand Sanadi** (University of Copenhagen);
12. **Dana Deselnicu** (Polytechnic University of Bucharest);
13. **Vendelino Neto** (TNS Antimicrobial Solutions);
14. **Holmer Savastano Junior, Juliano Fiorelli, João Adriano Rossignolo** and **Elíria Pallone** (University of São Paulo).

Managing and mentoring

1. **Chiara Davalli** (EBN Innovation Network);
2. **Luciana Lenhari** (UNICAMP)
3. **Laura Rivero** (CSIC - Spanish National Research Council)
4. **Paloma Tejedor Jorge** (CSIC - Spanish National Research Council)

Other invited participants

1. **Florindo Gaspar** (Polytechnic Institute of Leiria)
2. **Bhavna Sharma** (University of Bath, UK)
3. **Khosrow Ghavami** (PUC Rio)
4. **Roselena Faez** (Federal University of São Carlos)
5. **Ernesto Villar Cociña** (Universidad Central de las Villas)

Annex 3- List and Profile of registered participants

Name	email	Occupation	Institution
Adriana de Campos Pastre	dridecampos@yahoo.com.br	Professora	UFSCar
Adriana D. Costa Rocha de Sá	adrianadominique@gmail.com	Engenheira Civil	USP
Allan de Amorim dos Santos	allandeamorim@hotmail.com	Estudante	UFLA
Breno Guimarães Oliveira	florestal.breno@gmail.com	Estudante	UFLA
Carlos Alexandre Fioroni	carlosfioroni@usp.br	Estudante mestrado	FZEA-USP
Christian Gauss	gausschr@usp.br	Doutorando	FZEA-USP
Danilo Bordan Istuque	daniloistuque@gmail.com	Estudante de Doutorado	UNESP
Débora França	deboraf@usp.br	Doutorando	FZEA-USP
Erika Yukari Nakanishi	tyou.eyuna@gmail.com	Zootecnista	FZEA-USP
Guilherme Fernando Carmello	carmello.gf@usp.br	Estudante de Mestrado	FZEA-USP
Holmer Savastano Junior	holmersj@usp.br	Professor	FZEA-USP
Jordão Cabral Moulin	jordao_cm@hotmail.com	Estudante	UFLA
Jorge Luis Akasaki	jorge.akasaki@gmail.com	Professor Universitário	UNESP
Lais Kohan	laiskohan@hotmail.com	Estudante	EACH – USP
Larissa Oliveira Duarte	larissaoliveiraduarte@hotmail.com	Estudante	EACH – USP
Leticia Missiatto Gavioli	leticiamissiattoGavioli@gmail.com	Estudante (mestrado)	FZEA-USP
Lívia Ribeiro Costa	liribeirocosta@yahoo.com.br	Estudante	UFLA
Lucas Luiz Messa	messalucas@usp.br	Estudante (mestrado)	FZEA-USP
Luiz Eduardo Silva	lesilvaflorestal@gmail.com	Estudante	UFLA
Maria Luiza Cafalchio de Oliveira	luizacafalchio@gmail.com	Estudante	UFLA
Matheus Cordazzo Dias	matheus.cordazzo@gmail.com	Estudante	UFLA
Matheus Roberto Cabral	matheusr18@hotmail.com	Estudante doutorado	FZEA - USP
Murilo Alves Lima	murilo.alves.lima@usp.br	Estudante	USP
Patricia das Neves A. Santana	patriciadn@usp.br	Estudante Dr.	FZEA-USP
Rachel Passos de Oliveira Santos	rachelpassos@gmail.com	Química, Pos-Doc	FZEA-USP
Ronaldo Soares Teixeira	ronaldostx@gmail.com	Pesquisador, Pos-Doc	USP
Roselena Faez	faez@ufscar.br	Professora	UFSCar
Adhemar Watanuki Filho	adhemarwatanuki@yahoo.com.br	Engenheiro Civil	UNESP
Alisson Farley Soares Durães	alissonfarley91@yahoo.com.br	Estudante	UFLA
Camila Gruber Chiaregato	camilachiaregato@gmail.com	Aluna de graduação	UFSCar
Tamires S Pereira	tamicnt@usp.br	Aluna de Mestrado	FZEA-USP
Hélio Marcos Della Torre Barbosa	dtorrebarbosa@gmail.com	Estudante	FZEA-USP
Luciano Fonseca Pinheiro	lucianofpinheiro@ig.com.br	Estudante	USP- EACH
Gabriela Contieri	gabicontieri@hotmail.com	Estudante	UFSCar
Lays Camila Matos	laysmatos9@hotmail.com	Estudante de Doutorado	UFLA
Vivian Lara	vivianlara@usp.br	Professora	FZEA-USP
Jamile Raquel Regazzo	jamile.regazzo@usp.br	Estudante	FZEA-USP
João Ricardo Shibata de Barros	joaoricardosk8@gmail.com	estudante	UFSCar
Amanda Garcia de Oliveira	amanda.garcia.oliveira@usp.br	Estudante	FZEA-USP
Carlos Bonfanti	mclmgh@gmail.com	Engenheiro/Empresário	CARLOS BONFANTI- EIRELI

Viviane Correia	vivianecostcor@usp.br	Pesquisador, Pos-Doc	FZEA-USP
Loic B. Rodier	rodierloic@gmail.com	Pesquisador	FZEA - USP
Marzieh Kadivar	kadivaroo.yzduni@gmail.com	Estudante Dr.	FZEA-USP
Juliano Fiorelli	julianofiorelli@usp.br	Professor	FZEA-USP

Annex 4- Script of laboratory practice

Experimental Procedures by Dr. Giberto Siqueira, EMPA

TEMPO-Mediated Oxidation of Cellulose Fibers: The TEMPO mediated oxidation procedure of cellulose fibers is performed after the method described by Saito, et al.¹. The cellulose fibers are suspended in water in order to form a suspension with a concentration of **2% (w/w)**. TEMPO and sodium bromide (NaBr) are dissolved in water to concentrations of 0.1 and 1.0 mmol per gram of cellulose pulp, respectively, and mixed with the fiber suspension. The pH of the suspension is adjusted to 10 with NaOH solution (1 mol L⁻¹). A concentration of 10 mmol NaClO is chosen per gram of cellulose pulp. The TEMPO-oxidized cellulose fibers are thoroughly washed until the conductivity was similar to that of distilled water.

Fibrillation of TEMPO-oxidized Cellulose Fibers: The oxidized and purified cellulose fibers are dispersed in water to a concentration of 2% (w/w) and ground using a Supermass Colloider (MKZA10-20J CE Masuko Sangyo, Japan) to obtain cellulose nanofiber suspension. The degree of carboxylation of the obtained TO-NFC is determined *via* electric conductivity titration as described by Saito and Isogai² and Katz, et al.³.

Degree of Oxidation: The degree of oxidation (DO) referring to the average number of carboxyl groups per anhydroglucose unit is calculated using the following equation⁴⁰:

$$DO = \frac{162*(V_2-V_1)*c}{w-36*(V_2-V_1)*c} \quad (1)$$

in which V_1 is the volume of NaOH added for the neutralization of the strong acid, V_2 the volume for the neutralization of the weak acid, c is the concentration of NaOH (molL^{-1}) and w is the dry weight of the cellulose nanofibers (mg).

References:

1. Saito, T.; Kimura, S.; Nishiyama, Y.; Isogai, A., Cellulose nanofibers prepared by TEMPO-mediated oxidation of native cellulose. *Biomacromolecules* **2007**, 8, 2485-2491.
2. Saito, T.; Isogai, A., TEMPO-Mediated Oxidation of Native Cellulose. The Effect of Oxidation Conditions on Chemical and Crystal Structures of the Water-Insoluble Fractions. *Biomacromolecules* **2004**, 5, 1983-1989.
3. Katz, S.; Beatson, R. P.; Scallon, A. M., The determination of strong and weak acidic groups in sulphite pulps. *Sven. Papperstidn.* **1984** 87, 48–53.