

## Research secondment of Assist. Prof. Antoneta Tomljenovic, Ph.D., at the LEITAT, Terrassa, Spain

Tuesday, 12 April 2011

Antoneta Tomljenovi , Ph.D., employed as assistant professor at the University of Zagreb, Faculty of Textile Technology, Department of Materials, Fibres and Textile Testing, spent her research secondment within FP7 T-Pot project at the new premises of LEITAT Technological Center in Terrassa (Barcelona), from 28th February 2011 to 18th March 2011.

LEITAT is a Technological Center, member of TECNIO and recognized by the Ministry of Science and Innovation, that aims to collaborate with companies and institutions by adding technological value both to products and processes, and focuses its activity on research, development and industrial innovation (R+D+2i). As Technological Partner, the Center is clearly committed to adaptation to transform the technological challenges into economic and social value. Since its foundation in 1906, LEITAT has prioritized its vocation of proximity by strengthening the principles of professionalism and respect to people and environment at the same time. Since 1914 LEITAT has been located on Passeig 22 de Juliol and at present it is located in the Parc Científic i Tecnològic de Terrassa (Orbital 40) where they continue their work in this new headquarters building which is expected to meet the specific needs of a Center such as LEITAT: sustainability, technological innovation, functionality, environmental quality and integration in the environment.

- a.
- b.
  
- c.

LEITAT: New headquarters building

### LEITAT RELIABILITY

#### Certifications of the Center

.  
Quality management  
system (standard UNE EN ISO 9001: 2000)

.  
Environmental  
management system (standard UNE EN ISO 14001: 2004)

.  
R+D management system

(standard UNE 166002: 2006)

.

Collaboration to the  
Community system of management and environmental audit (EMAS) Governed by  
Regulation EC No 731/2001

Accreditations  
of the Center

Quality  
management system (standard UNE EN ISO/IEC 17025: 2005), to the following  
technical dossiers

.

Wool and textiles  
products (18/LE026)

.

Behavior to burning  
(18/LE274)

.

Personal protective  
equipment PPE (18/LE705)

Self-Validation  
of RENAULT for testing parts of the interior and exterior as well as mechanical  
and electrical components

Laboratory  
recognized by the International Federation of Automobile for testing on  
protective clothing and gloves for pilots according to standard FIA 8856 - 2000

## LEITAT RESEARCH UNITS

From a traditional structure to a flexible and dynamic organization, LEITAT seeks to ensure a rapid and effective response by reducing levels of decision-making and bureaucracy. Prioritizing teams work and projects with a horizontal communication and a definition of responsibilities in order to achieve meet the expectations of the client and society.

## Biomed

R  
& D into new drugs aimed at cancer therapy, generation of monoclonal antibodies customized, Identification of new therapeutic indications for drugs, design of biosensors and diagnostic kits.

## Industrial Biotech

R  
& D in biotechnological solutions (microorganisms and enzymes) with application in industrial processes; Development and study of the effectiveness of antimicrobial, antifungal and dust mite.

## Nanomaterials

Customized  
preparation and characterization of new nanomaterials, nanofibers and nanofluids with new advanced properties for application in several industries.

## Nanosecurity

Assessment  
of risk to human health and the environment from the use of nanomaterials in products, R & D production methods and recycling of nanomaterials.

## Surface Treatments

R  
& D and monitoring specific functionalization of surfaces with new advanced technologies, optimization of formulations and industrial finishing processes

## Smart Systems

R  
& D in new concepts of intelligent systems based on sensors - actuators; industrial application of the thermoelectricity; new generation and energy transfer methods.

## Renewable Energies

R  
& D on new solar cells and solar concentrators, R & D on thermoelectric materials for implementation of Energy Harvesting; biopiles and mass storage Biorefinery.

## Environment

## Treatment

and Minimization of industrial water consumption for advanced technologies, energy efficiency studies and life cycle analysis.

## Textile Technologies

### R

& D on new textile materials and textile applications through a full investigation from fibres to clothing, technical textiles, personal protective equipment and smart electronics integration.

## Advanced Polymers

### Preparation

of new polymers and biopolymers. R & D in composites and nanocomposites for high performance and sustainable. Studies of recyclable and new applications.

## Fast Moving Consumer Goods

### R

& D aimed at adding value and sustainability for FMCG products. Methods and technologies for the treatment, protection, maintenance and improve of different products in the domestic and industrial sphere e industrial.

## Analytical Chemistry

### Development,

validation and application of new analytical methods. Studies of emerging pollutants. R & D aimed at the recovery of waste and materials from renewable sources, preparative and industrial chemistry Replacing highly dangerous chemical reagents.

## New Production Processes

### (R+D)

and industrial support in new additive manufacturing processes. Design and adequacy of industrial processes, productive tools and prototypes; Manufacturing processes for obtaining new products of high performance

## FIELDS OF KNOWLEDGE & MARKET

### Today the transformation

of the LEITAT is continuous and draws a new model for the future, enabling them to provide greater technological value long-term to their customers that enable them to maintain their principles and values. From the collaboration they harness synergies between their units research bringing a more critical mass to deal with risk projects.

They

work to be ahead and transform the technological challenges and needs companies and institutions face regarding their high value products and processes, satisfying the market's changing needs. Their mission is to transfer knowledge to their clients, while guiding them and conducting a follow up of the challenges they propose, achieving at the same time the consolidation of the four working areas of the center:

.  
R+D Transference (applied, competitive, and collaborative research)

.  
Advanced Technological Services (testing, material behavior under extreme conditions and according to the regulations)

.  
Notified Body (homologation and certification of products according to specifications and regulations)

Innovation and new technologies (management models and tools to foster corporate innovation)

STAY OF DR. ANTONETA TOMLJENOVIC IN LEITAT

.  
Person  
In-Charge: Dr. Roshan Paul, Principal Investigator of Textile Technologies (TTE) Division

.  
Objective:

- To carry out research activities for developing Multifunctional Protective Uniforms. This includes the chitosan microcapsule/microsphere development for its incorporation on textiles. A second part involves incorporation of thermochromic pigments inside PLA polymer by extrusion.

- Additionally to learn about ECO-Label, LCA, Textile R&D and Textile Testing.

Working plan:

A. Plan for the week 28/02/2011 to 04/03/2011

DATE

TASKS

IN-CHARGE/ REMARKS

28/02/2011

Introduction  
to TT activities and general themes

Roshan  
Paul

01/03/2011

Development of chitosan microcapsules /microspheres by suspension crosslinking

Anna Surribas/Laia Crespo Solana

02/03/2011

Development  
of chitosan microcapsules /microspheres by suspension crosslinking

Anna  
Surribas / Laia Crespo Solana

03/03/2011

Development  
of chitosan microcapsules /microspheres by suspension crosslinking

Anna  
Surribas / Laia Crespo Solana

04/03/2011

Development  
of chitosan microcapsules /microspheres by suspension crosslinking

Anna  
Surribas / Laia Crespo Solana

B. Plan for the week 07/03/2011 to 11/03/2011

DATE

TASKS

IN-CHARGE/ REMARK

07/03/2011

Review  
of previous week, introduction to TTE activities and general themes

Roshan  
Paul

08/03/2011

Incorporation of thermochromic  
pigments in PLA polymer by extrusion



Mercè  
de la Fuente Jordà

09/03/2011

10/03/2011

11/03/2011

Injection of thermochromic pellets into plaques

Mercè de  
la Fuente Jordà

C. Plan for the week 14/03/2011 to 18/03/2011

DATE

TASKS

IN-CHARGE/ REMARKS

14/03/2011

Review  
of previous week, introduction to TTE activities and general themes

Roshan  
Paul

15/03/2011

ECO-Label,  
LCA and other certifications

Marta  
Escamilia Monell

16/03/2011

17/03/2011

Textile  
laboratories: R&D, testing and characterization

Helena  
Esteve/ Roshan Paul

18/03/2011

General  
review of the stay, conclusions and future plans of collaboration.

Roshan  
Paul

## RESEARCH REPORT

Outline of the prescribed work:

The general goal for the project is to develop protective uniforms, incorporating multiprotective properties. The protective uniforms will be able to protect

against the multiple hazards of risky situations: protection against wetting and water permeation; protection against extreme environment temperatures; protection against microbial contamination; protection against fire and associated heat; protection against static electricity.

#### A. Development of chitosan microcapsules/microspheres by suspension crosslinking

##### Microencapsulation

•

##### Microcapsules

are micrometer-sized particles formed by an external thin spherical polymeric shell covering an active ingredient found in the interior (core), due to the antimicrobial action and the polymeric nature of the chitosan microcapsules can be formed exclusively of chitosan to form both the core and the shell.

•

Main objectives of microencapsulation are:

- Immobilize a compound
- Protect it of external agents
- Controlled release

##### Methods of microencapsulation

#### 1. Chemical methods:

•  
Interfacial polymerization:  
Characterized by wall formation via the rapid polymerization of monomers at the surface of the droplets or particles of dispersed core material.

•  
Coacervation method:  
MCs are formed by interionic interaction between oppositely charged polymers.

.  
Ionotropic gelation:

Driven by a syringe pump, the chitosan solution flowed out of the hollow needle and was divided into liquid droplets by the high voltage electrostatic force that reacts with coagulation solution forming MC.

2. Physical  
methods:

.  
Spray-drying:

Chitosan solution is sprayed, air-dried followed by the addition of a crosslinking agent. The preparation parameters with the spray dryer technology (size of nozzle, feeding pump rate, inlet temperature, and compressed air flow rate) influence the particle size of the microsphere. Is a versatile and economical method to encapsulated compounds.

Spray dryer

Microcapsule

Cross-linking  
agents

Chitosan can  
be cross-linked either ionically or covalently:

&bull;

Ionic cross-linkers: Polyanions  
such as triphosphate (TPP), citrate or natural polymers like hyaluronic acid or chondroitin sulfate.

&bull;

Covalent cross-linkers: A

covalent network could be performed if the amino or the hydroxyl groups of chitosan react with functional groups of the cross-linker molecules. Examples of covalent cross-linkers: di- or polyaldehydes (glutaraldehyde) oxidised starch and oxidised cyclodextrin, carboxylic acids, azides, epoxides, diisocyanates and silanes.

Chitosan microspheres preparation by suspension crosslinking

Research activities

The antibacterial functionalization of textiles will be addressed through the development and application of chitosan microspheres/microcapsules. Chitosan microspheres/microcapsules may provide antibacterial properties. It contemplates the development of chitosan microcapsules or microspheres using the suspension crosslinking method and their subsequent application on textiles. Following the suspension crosslinking method, an acetic aqueous of chitosan is initially prepared and dispersed in a continuous organic phase. Subsequently, the crosslinking agent is added to the dispersion and the precipitated crosslinked microspheres are recovered. Different types of bifunctional agents (glutaraldehyde and triphosphate) are used for this purpose.

The crosslinked microspheres have the advantage of being insoluble in all solvents, however if the crosslinking is performed through chitosan's amine groups (e.g. using glutaraldehyde as a crosslinker), attention is required so as to not fully crosslink the network. This is very important in order to maintain free amine groups, as these provide to chitosan its antibacterial properties. The application of higher mixing speeds or the use of homogenizers may allow for better phase dispersion during microparticles preparation and thus lead to the preparation of smaller particles.

Chitosan  
microspheres crosslinked with glutaraldehyde

We have obtained chitosan  
microsphere powder by the suspension crosslinking method. We have tested two  
different crosslinking agents - glutaraldehyde which causes covalent  
crosslinking through amine groups and tripolyphosphate responsible of the ionic  
crosslink through protoned amine groups. In case of glutaraldehyde  
crosslinking, the color of the powder is directly related to the degree of  
crosslinking (volume of crosslinking agent used).

Chitosan micro-  
spheres preparation  
Microscopic laboratory

Dispermat

Microbiological laboratory  
Chemical laboratory

## B. Incorporation of thermochromic pigments in PLA polymer by extrusion

### Research activities

Monofilament yarns were prepared from the thermochromic masterbatches by melt spinning. The processing conditions were optimized through a series of trials on the temperature and the velocity of the melting (extrusion) process and the tension of elongation. The main objective was to obtain uniform and resistant yarns avoiding the degradation of the chromic material during the melt-spinning process. The % dilution of the masterbatch was also studied. PLA monofilament yarns with a diameter in the range of 0.1 to 0.4 mm were obtained with adequate color change properties.

Antoneta  
Tomljenovi

Roshan  
Paul and Antoneta Tomljenovi



Angel  
Garcia Roman

High  
security measures during the work

Twin screw extruder with polymerization reactor + melt spinning pellets  
unit or melt spinning monofilament unit

PLA  
pellets production

PLA  
pellets + yarns

## C1. ECO-Label, LCA and other certifications

The Environment Unit of LEITAT

Technological Center participates in regional, national and European projects regarding environment and sustainability. The main fields of activity are the following:

### Industrial Ecology

Sustainable combination of technology, environment and economy. Assimilation of the function of industrial areas to natural ecosystems with more efficient processes and with an interrelation between industries and environment closing material cycles.

- 
- 
- Energy Efficiency: Energy Analysis of processes, installations and buildings
- 
- Water: Consumption reduction, management, treatment and reutilisation of waste water
- 
- Waste (prevention, minimization and recycling): Utilization of waste at the same industry, studies on waste minimization, feasibility study on the reuse of generated waste.
- 
- Sustainable Mobility: sustainable mobility studies
- 
- REACH, CLP: Registration, evaluation and authorization of chemical substances, classification and labelling.

### Ecodesign

Incorporation of environmental criteria into the design of a product/service in order to reduce the negative impact on the environment.

- 
- Life Cycle Analysis (LCA) LCA of a product, a service...
- 
- Ecodesign of a product, a production process, a package...

-  
Carbon Footprint

-  
Green procurement

-  
Greening events

### Awareness and Environmental Education

Good environmental training helps to improve business competition, Corporate Identity and the relationships between interested groups.

-  
Best Environmental Practices:  
Consume reduction, Waste minimization ...

- Environmental awareness campaigns  
Waste reduction campaign, resources minimization...

-  
Green procurement and events:  
Sustainable events ...

- Training sessions and Seminars.  
Environmental Management, Ecodesign, Life Cycle Assessment, Best Available Techniques, Energy Efficiency, Social Responsibility, Good environmental practices, REACH, CLP...

### Environmental Management and Qualification

Voluntary instruments which enable the organization to reach a high level of environmental protection and a compromise on a continuous improvement.

-  
Environmental Management Systems  
(EMS) in accordance with the EU Regulation EMAS and the International standard ISO 14001

-  
Energy Management Systems UNE 16001

, Ecodesign management UNE 150.301

- Social Responsibility Plan for equal opportunities between men and women, court settlement f work and family life, EFR, SGE21...

- Ecological Label of the European Union (Ecolabel)

- Emblem of Guarantee of Environmental Quality of the Catalan Government (DGQA)

- Environmental product declarations (EPD)

LEITAT Technological Center is an associate body of the Catalan Government with the goal to carry out tests, controls and verifications of evaluation results of the following product/service categories:

Emblem of Gaurantee of Environmmmental Quality European Union

- o Water Saving Systems
- o Recyclable products made of cardboard
- o Rural tourism establishment
- o Service and delivery stations
- o Establishment of a perfect photo finishing
- o Products made of brushwood
- o Dry cleaner's
- o Dry recycling products
- o Customer Services Offices

o Office Buildings

Eco Label of European Union

o Textile Products

o Laundry Detergent

o Soil Amendments

o Cultivation Substrates

o Interior paints and varnish

o General cleaning products

o  
Soaps and Shampoos

Members of the Environment Unit of LEITAT  
(Gertri Ferrer Vinardell, Marc Torrentellé Soler, Natalia Fuenes Cortés, Raquel  
Villalba Gonz les) and Antoneta Tomljenovi

C2.Textile laboratories: R&D, testing and

## characterization

## R&amp;D

Textile Technologies Division works in different textile subareas from traditional textiles to the new technologies and trends of the market. The research activities of the Division include new fibres, spinning, weaving, textile finishing, technical textiles, PPEs and smart textile structures.

Fibres: Division works with all

kind of fibres; Study of innovative and sustainable processes

Spinning: Expertise in the spinning sector

Knitting and weaving with

conventional fibres or with modified and new fibres

Dyeing and finishing:

Microencapsulation (microcapsules development with new properties, microencapsulation, application on textile substrates); Nano-finishing (Ultraviolet protective

textiles, Lotus effect textiles, Self cleaning textiles); Eco-treatments

(Anti-odour textiles, Antimicrobial textiles, Flame retardant nonwovens, Textiles

dyed with natural mordants and dyes, Textile dyeing incorporating hydrolyzed protein, Optical brightening incorporating hydrolyzed protein);

Bio-finishing (Cotton bio-finishing; Hydrophilic and easy-care bio finishing); Special pigments (Thermochromic textiles, Photochromic textiles, Phosphorescent textiles, Optically variable textiles)

Smart Textiles (Electronic

parts in textiles; Intelligent materials)

Personal Protective Equipments (different application fields)

Technical textiles (different application fields)

a. Printing equipment - application

of phosphorescent pigments

b. Electrolux Wascator FOM71LP-lab

c. Finishing powder spray equipment

d. Laundry

e. ATLAS Xenotest ALPHA

f. Salt spray cabinet

LEITAT laboratories: a. Marolda Brouta-Agnésa and Antoneta Tomljenovi , b., c. Antoneta Tomljenovi , d. Ester Delgado, e. Marolda Brouta-Agnésa, f. Provi Ortiz

## CONCLUSION

The overall aim of the FP7 Project-REGPOT-2008-1: Unlocking the Croatian Research Potentials (T-Pot) is to strengthen the scientific potential of the Faculty of Textile Technology of University of Zagreb -in that will allow greater inclusion of the Croatian textile organizations in the research activities at European level and thus support the process of harmonization and integration of European research (European Research Area - ERA). With purpose of mobilization of human resources and given the current trend of lifelong education and the necessity of permanent adoption of

new knowledge and skills, the T-Pot project organize shorter visits of Croatian researchers in European institutions, and vice versa, for the purpose of the transfer and application of new knowledge. We hope that stay of Antoneta Tomljenovi in LEITAT Technological Center, Spain will result with good future cooperation between our two institutions and scientists primarily through joint publications and projects.

a

b

c

d



e

f

Comfortable LEITAT work areas: a. Roshan Paul and Antoneta Tomljenovi , b. Roshan Paul, Lorenzo Bautista and Antoneta Tomljenovi , c. Anna Surribas, Laia Crespo Solana and Antoneta Tomljenovi , d. Antoneta. Tomljenovi ; and LEITAT restaurant: e. Roshan Paul, Moisés Moron and Antoneta Tomljenovi and f. Laia Crespo Solana, Mercè de la Fuente Jordà and Helena Esteve

## ACKNOWLEDGEMENT

I, Assist.

Prof. Antoneta Tomljenovi , Ph.D. would like to thank Prof. Sandra Bischof Vukušići , Ph.D. (FP7 T-Pot European project coordinator) for professional support. I would like to express special thanks to Dr. Roshan Paul, Principal Investigator of Textile Technologies Division, LEITAT, for professional and friendly help. Additionally I would like to thank Mrs. Meritxell De la Varga Ortiz, Manager, R&D Department for professional support and all the employees of LEITAT who provided my pleasant stay in LEITAT.

Download complete report (same as this web page) in WfW doc format:

Report\_Leitat\_Antoneta Tomljenovic\_Public.doc