

PROJECT FINAL PUBLISHABLE REPORT

Grant Agreement number: **228730**

Project acronym: **SONO**

Project title: **A pilot line of antibacterial and antifungal medical textiles based on a sonochemical process**

Funding Scheme: **FP7- NMP-2008-1.2-1**

Date of latest version of Annex I against which the assessment will be made: **03.02.2010**

Period covered: from **October 1st, 2009** to **September 30, 2013**

Name, title and organisation of the scientific representative of the project's coordinator:

Prof. Aharon Gedanken, Bar-Ilan University - BIU

Tel: **+972 3 5318315**

Fax: **+972-3-535-1250**

E-mail: **gedanken@mail.biu.ac.il**

Project website address: **<http://www.sono.eu/>**

Final Publishable Report

4.1 Final Publishable t Summary

The idea behind the SONO project was an attempt to make the Hospital a safer place by replacing all the currently used textiles by antibacterial textiles. It is well-known that approximately 1 million people are dying every year in Hospitals around the world out of nosocomial infections.

SONO was based on early results demonstrating that metal oxide (MO) nanoparticles (NPs), (ZnO, CuO, and MgO) sonochemically coated on various fabrics can kill efficiently a large variety of bacteria.

It was therefore proposed that a step forward towards the goal of eliminating nosocomial bacteria, machines for a roll to roll sonochemical coating will be built and the coated fabrics will be checked for 1) antibacterial properties, 2) mechanical properties 3) leaching into water and saline solution and 4) withstanding long washing cycles at Hospital washing machines (75 and 92 0C). These properties were in addition to regular characterization measurements for determining the amount of the NPs on the fabric, the size and shape of the NPs.

Two roll to roll sonochemically coating machines were built in the course of the project. Both have produced a homogeneously nice coating of the MO NPs on cotton and Polyester and their mixtures. The coated fabrics revealed excellent antibacterial properties. No leaching of NPs of MO into water or saline solution is detected over 96 testing hours.

The coated fabrics were washed 65 cycles at 75 0 C using a pH neutral detergent and were found having a $> \log 4$ killing of a few common bacteria.

Unforeseen achievement in the course of the project

A new coating sonochemical technique called "throwing stones" was developed in the course of the project. It resulted from the industrial complain on the use of ethanol in the synthesis of the MO NPs. The ethanol was avoided by purchasing MO NPs from a commercial source and employing the ultrasonic waves to throw the NP onto the fabric.

The partners have discovered that the MO NPs can kill not only sensitive bacteria but also resistant bacteria, i. e. bacteria resistant to antibiotic.

Finally the answer to the question can the Hospital become a safer place using the MO NPs was given in an experiment carried out in the PIGOROV Hospital in Sophia Bulgaria in an experiment testing 25 patients in an rooms fully equipped with antibacterial coated NPs.

THE ANSWER WAS YES, THE HOSPITAL IS SAFER FOR PATIENTS USING THE SONO FABRICS.

It is therefore proposed that the next step should be building an industrial machine answering specific requirements of an end user

Final Publishable Report

Description of project context and objectives

Concept

Hospital-acquired (nosocomial) infections are a major financial issue in the European healthcare system. The financial impact of these infections counteract medical advances and expensive medical treatments by increasing the length of hospital stay by at least 8 days on average per affected patient, hence adding more than 10 million patient days in hospitals in Europe per year. The statistics on patient safety in the EU show alarming tendencies:

- 1 in 10 patients are affected by hospital-acquired infections
- 3 million deaths are caused by hospital-acquired infections yearly

An active infection control program of patients and personnel and hygiene measures, have proven to significantly reduce both the number of infections and hospitalisation costs.

The SONO project directly addresses the above problems by developing a pilot line for the production of medical antibacterial textiles.

The pilot line will be based on the scale-up of a sonochemical process developed and patented at BIU laboratories. The pilot line will use a sonochemical technique to produce and deposit inorganic antimicrobial nanoparticles on medical textiles, e.g. hospital sheets, medical coats and bandages.

Sonicators are used industrially for heavy and light duty cleaning, for water disinfection and for sewage treatment. It is also used in the food industry for emulsification and drying. The proposed concept based on one step sonochemical continuous process to produce nanoparticles and at the same time impregnate them as antibacterial factors on textile is novel in an industrial scale for this application.

The concept has already been proven and patented on a lab scale where sonochemistry was applied to impregnate nanoparticles in a single-step process.

It was demonstrated that due to the special properties of the sonochemical method the antibacterial nanoparticles are adsorbed permanently on the fibres even in the course of 70 "laundry cycles" as proven by BIU. The sonochemical impregnation process is a one-step procedure in which the nanoparticles are simultaneously created in solution and applied onto the fabric surface at such a high speed that they either form chemical bonds with the textile substrate, or physically embedded in the fabric in cases where no functional groups are available in the fibre (i.e. polypropylene).

Thus the proposed process has its main advantages in: reducing production time due to the very effective one step process, reduction of fibre damage, increasing the uniformity of distribution of the nanoparticles, lowering product cost (based on Mg or Zn oxides versus silver for existing products) and environmentally friendly because it uses water solution, and there is no leaching of the nanoparticles to the environment.

Objectives

The main objective of this project is to build a pilot line based on a sonochemical reactor to produce biocidal textiles by impregnating antibacterial nanoparticles (e.g. MgO and ZnO) on the fabrics. The proposed pilot will be based on modeling of sonochemical reactors. The process will be metrological and safety controlled.

Final Publishable Report

The process will lower product cost (based on Mg or Zn oxides versus silver for existing products and Ultrasound reaction – a low cost process); it is safe (no subsequent reversal or loss of ZnO/MgO to the patient wound) and environmentally friendly using water solution and low amounts of metal oxides (Zn and Mg); reduce the production time (one step) ; reduce the fibre damage, efficient ; increase nanoparticles distribution uniformity,.

1 Design and characterization of the pilot line and products

- 1.1 Pilot components identification and specification/ design
- 1.2 Metrology, monitoring and safety devices identification and specification/ design
- 1.3 Characterization and design of workers and environmental protection
- 1.4 Product Characterization
- 2 Develop modeling for sonochemical reactors
 - 2.1 Electro-acoustic modeling
 - 2.2 Modeling acoustic waves in bubbly liquid
 - 2.3 Modeling of mechanical mixing in the reactor
 - 2.4 Modeling multiple bubble dynamics
 - 2.5 Modelling of transport of chemicals in the reactor
 - 2.6 Modelling of heat transfer in the reactor
 - 2.7 Optimization of the sonochemical reactor

3 Development of metrology, online monitoring, safety and control system for the Pilot line - SW and HW

- 3.1 Metrology, Monitoring, Safety and Control application requirements definition
- 3.2 Design of Metrology, monitoring and control system
- 3.3 Develop application software for data acquisition, on-line monitoring and automation
- 3.4 Develop application software for offline analysis and correlation with test results.
- 3.5 Integration and testing
- 4 Pilot line components manufacturing and assembly
 - 4.1 Production of the pilot line components
 - 4.2 Assemble the pilot lines, at end users' sites
 - 4.3 Analysis of the two pilot lines performance and optimization of one final pilot
 - 4.4 On-going support of the pilot line operation

5 Optimization of the pilot processes (antibacterial activity) and production of the antibacterial textiles

- 5.1 Optimisation of Ultrasonic process parameters and manufacturing of the products accordingly
- 5.2 Optimization of the textile products with simultaneous sonochemical/enzymatic/NPs coating of textiles and manufacturing of the products accordingly.
- 5.3 Simultaneous hybrid (biopolymer-NPs) antimicrobial embedment using US and manufacturing of the products accordingly

6 Product Characterization and end-user validation

The main objective here will be to get the highest values for reproducibility, accuracy and precision.

- 6.1 Development of a web site for on line data acquisition of samples characterization
- 6.2 Physical - Mechanical and chemical analysis of the textiles
- 6.3 Assessment of the antibacterial efficiency and antifungal properties of the biocidal textiles
- 6.4 Tests the leaching of the nanoparticles from the textile to the environment
- 6.5 Characterization and production of confection methods and production of pillow covers, bad sheets and pajamas with the antibacterial textiles
- 6.6 Product validation by the end users

Final Publishable Report

6.7 Conclusions of product validation by the end users

7 Study the economical environmental and toxicological impact of the process developed

7.1 Process and product cost effectiveness for the pilot and the up scaled design - LCC

7.2 Life Cycle Assessment (LCA) of product and processes following ISO 14040 standards, according to SETAC proposals

7.3 Evaluation of the impacts of the new products on medical textiles regulations

7.4 Market analysis and economic impact.

8 Design, modeling and evaluation of a scaled up (X5) industrial production line

8.1 Define envisaged obstacles for the scaled up process

8.2 Design and characterization of a scaled up production line

8.3 Evaluation of investment, time to the market and product cost

Final Publishable Report

4.2 Main S&T results/foregrounds

Modelling of sonochemical reactors (WIT)

This work package provides tools for optimization of operating regime/conditions for the sonochemical reactor. The main objectives/tasks in this work package are explained below.

2.1: Establish the relations between the voltage/current applied to the ultrasonic transducer and the in-fluid acoustic field versus frequency

The wave propagation on the fluids depends on the US generator "Transducer & resonator" dimensions. On the CED US generators, the transducers are connected to the tubular resonators. The resonator deformation shape and the corresponding frequency resonances depend on the length of the resonator. The results have been presented in the deliverable D2.1.

2.2: Define the volume of the reactor where conditions for cavitation exist taking into account interaction of bubbles with acoustic field

A mathematical model to simulate the acoustic wave propagation during the operations of the transducers was developed. The effects of cavitation bubbles were included by introducing a complex wavenumber to the governing Helmholtz equation. An in-house code based on the meshless radial basis integral equation method has been developed. A sensitivity analysis was carried out to examine the effects of various parameters on the pressure distribution inside the tank. With the in-house codes, zones where the cavitation threshold is exceeded can be prescribed with the complex-valued wavenumber while zones where the pressure is lower than the cavitation threshold can be prescribed with the real-valued wavenumber. Details of the models and results for the acoustic waves in bubbly liquid can be found in the deliverable D2.5 .

2.3: Design the mechanical mixing required for efficient operation of the reactor and delivery of the required chemicals to the cavitation zone

A numerical model for transport of chemicals in the sonochemical reactor has been developed. The model requires as an input the velocity field which is obtained as a result of the flow model. The model for transport of chemicals can be used for analysis of the transport and reactions in the sonochemical tank, and also to design a better mechanical mixing if required. The initial reagent added to the reactor is $\text{Zn}(\text{OAc})_2$ and as solvent water-ethanol mixture is used. After the temperature reaches 60°C, ammonia solution (NH_4OH) is added to the reactor. The zinc hydroxide reacts further with ammonia solution yielding tetra ammonium zinc hydroxide (TAZH). Details of the models and results for mechanical mixing and transport of chemicals in the reactor can be found in the deliverables D2.2 and D2.3 , respectively.

2.4: Determine the heating requirements and viscosity of the liquid

A three-dimensional numerical model for the pilot plant designed by CED has been developed. The model is used to simulate the flow and heat transfer inside the tank before and during the operation of the heaters. Mechanical mixing is included into the model. It was found that the flow due to buoyancy is comparable or more significant than the flow due to mechanical mixing in the upper part of the reactor. The model was calibrated using temperature measurements in the reactor. Details of the model and the results obtained can be found in deliverable D2.4 .

2.5: Develop a model for multiple bubble dynamics in order to establish a link between the micro and macro processes/parameters in the reactor

A 3D numerical model of multiple bubble dynamics was developed based on the boundary element method. With the developed model, parametric study was undertaken analysing the bubble dynamics along a rigid wall. The effects of the following reactor parameters were investigated: wave amplitude and frequency, temperature of the liquid, liquid properties (density, surface tension and ratio of specific heat for vapour of the liquid and gas), initial bubble size and distance from the wall.

In ethanol, the bubble evolves faster and ends with higher jet speed, temperature and pressure than in water. The influence of ambient temperature on the bubble evolution becomes prominent when it is close to the boiling point of

Final Publishable Report

the liquid, yielding a higher jet speed during the bubble collapse. The analysis of effects of surface tension showed negligible when the initial bubble radius $R_0 \geq 10 \mu\text{m}$.

Simulations were also carried out on a cluster of bubbles of different sizes containing up to 100 bubbles. The effects of the bubble cluster on the potential field across the solution domain were investigated. The presence of a solid boundary was found to boost the interactions within a bubble cluster. A bubble cluster of different bubble sizes results in more turbulent flow, thereby potentially leading to more noticeable impacts on the solid boundary. More details on the developed models of bubble dynamics and obtained results can be found in deliverables D2.6 and D2.8.

2.6: Optimize the operating conditions/parameters in the reactor

This task utilized the developed models from the previous tasks to the optimization of the acoustic pressure distribution inside bubbly liquid and the mixing and transport in the sonochemical reactors of VIA and CED. An optimization based on the distance between the fabric and the transducer was performed. The results of the analysis suggest that the distances of 5 and 20mm offer more optimal operating conditions for the reactors of VIA and CED, respectively, although the accuracy of the results is subject to the accuracy of the assumption for the volume fraction and bubble sizes used. An approach to estimate the attenuation coefficient based on the calorimetric measurements was proposed, which will help with future simulations. The results indicated the possibility of uneven coating due to the nodes of the pressure waves between the left and right transducers almost coinciding with one another.

Simulations for the VIA reactor showed that the mixing in the zones where sedimentation occurs can be improved by using a 4-inlets, 2-outlets configuration appropriately placed around the walls of the tank. More details on the optimization of the operating conditions in the pilot plants can be found in the deliverable D2.7.

2.7: Design a scale-up for the reactor to a full scale plant

The scale-up of the prototype reactors developed by VIA and CED was carried out. It was decided that a direct scale-up of the reactor's dimensions was not feasible since the other parameters such as pressure, power input and the optimal distance between the fabric and the transducers do not necessarily scale linearly. For the VIA's reactor, two designs based on increasing the number of transducers were created. The first design employed 8 transducers and the contours of the pressure across the fabric appear to suggest non-uniform nanoparticles coating. To overcome this problem, the second design employed 12 transducers and they were placed so that wave nodes and antinodes from the transducers coincide.

A potential scaled-up design of CED's reactor was also proposed which showed that a scale up with the current design of the transducers may not be feasible. Therefore numerical simulations were performed for the scale-up design of the sonochemical reactor utilizing a new tubular resonators developed by CED and VIA. Trapezoidal reflectors were utilized in order to improve the efficiency. More details can be found in deliverables D8.2 and D8.3.

Metrology, online monitoring, and the control system development (AFI)

The Sonochemical machine implemented on two pilot lines, includes various process variables, parameters and actions which are required to be controlled by automatic system to ensure the proper conditions for impregnating antibacterial nano-particles into the desired fabric. The control system of the pilot line includes a software application with a dedicated HMI which will provide online display of the monitored process parameters. The HMI application activates manual and automatic control of the pilot line mechanism (pumps, valves, fabric progress). The system displays alerts for values out of valid range, and provides alarms for safety events. The application is based on the PULSE SCADA/HMI software which was developed for supporting the specific requirements of online application to control the 2 pilot line machine assembled during the SONO project.

The system designed includes a programmable logic controller (PLC) which controls the various end point devices as inputs/outputs to the main SCADA/HMI server and the logging database.

The application is integrated with the Offline Analysis tool used for evaluating the process operation parameters compared with the quality of the produced fabric (results of WP6), and allow tuning and optimization of the SONO process for different production scenarios.

SONO Online application – Process operation display

Final Publishable Report

During the project the work in WP3 covered the following activities:

Requirements definitions for monitoring and control HMI application, and for the offline process analysis tool, and were based on pilot machine definitions in WP1, which were elaborated in regard to process monitoring, control and automation of the SONO machine.

The requirements definition was summarized in D3.1 - Metrology, monitoring, safety and control application and process analysis tool requirements document.

Detailed design of metrology, monitoring and control system – including definition of process control and automation methods for the sono-chemical process, based on metrology and automation equipment installed in the SONO machines, in different scenarios of operation.

The Detailed design was formalized in D3.2- Detailed design of the metrology, monitoring and control system, which covered the Definition of metrology and control devices, the Online and Offline applications high level design, and Detailed Design for system modules.

The SONO machine operation was defined in three automated processes:

Reagent Preparation: Filling water and reagent in preparation tank and mixing to dissolve.

SONO tank preparation: Filling main tank with ethanol, Ammonia and reagent, activating mixing pumps and heating until target temperature is reached.

SONO process activation: Activating Sonotrodes and Fabric motor to start production. Adding Ammonia and reagent to compensate and maintain concentration.

Development of application software for data acquisition, on-line monitoring of sonochemical process parameters and automation of SONO machine operation. The application development was performed in several phases, following changes in the machine design and the process automation needs in the two SONO pilot machines.

Develop application software for offline analysis and correlation with test results: The offline tool can present comparison of different executions of the US process, as well as test results for the produced fabric . Data is taken from the SONO machine database, and from the samples characterization database maintained by PR.

Development of PLC software for retrieving process parameters and automatic operation of the SONO machine components, implemented separately for the two SONO machines.

Integration and testing of the control equipment, the PLC, and the SONO application software: The work included simulation of sensor data and validation of control system performance in the target scenarios. The integration tests were describes in D3.4 - Report on lab tests of the developed devices.

Offline Analysis application – Experiment Summary screen and Graph Results screen

The products of the above development was installed on the SONO machines in DAVO and KLO sites, and is currently under further testing and adjustments during last period of SONO project. In addition, the development of offline analysis application software will continue to support further experimentation with the SONO machine and optimization of the SONO process.

Summary of overall achievements:

- Design of the metrology, monitoring and control system for SONO machine
- Develop application software for data acquisition and on-line monitoring
- Construct monitoring control and automation system for both SONO machines
- Develop automation mechanism for SONO process
- Develop application software for offline analysis of test results

Pilot components manufacturing and assembly (CED)

The aim of this work package was to manufacture the sonochemical machine pilot line based on the recommendations and the design achieved on the WP1. The objectives of this work package are given here after.

- Producing the pilot line components
- Assemble the pilot lines, at end users' sites
- Analysis of the two pilot lines performance and optimization of one final pilot
- On gong support of the pilot line operation

Final Publishable Report

The functional schema of the chemical process as defined in the D1.1 is given here after. The numbers of tank requested for the chemical process are:

- The reagent tank: The chemical preparation before sonochemistry process.
- The sonochemical tank: The ultrasonic transducers are installed in this tank to generate the cavitations.
- The squeezing roller wasted: The chemical solution is collected after squeezing process.
- The ethanol recovery tank: The wasted ethanol vapor is collected in this tank.

The textile is unfolded on the sonochemical tank for coating process. The textile is unrolled near the ultrasonic transducer (tubular transducer for the CED machine and flat transducer for VIA machine). A drying machine is added at the end of the pilot line.

The machine has been manufactured and assembled at the CED facilities. The connection and the cabling of the whole machine have been done also at the CED facilities.

The list of sensors has been defined by chemists needed to control the chemical process. The automatic valves have been installed between the tanks for full automation control of the machine. The connection between all subsystems of the machine is presented here after.

Both pilot lines have been manufactured and are fully controlled

Both pilot lines (CED / VIA) were tested in three levels, component, subsystem and coupling configuration

The first test show (visually) the cavitation generation in the both pilots' line

The unfolding textile sub system has been tested on speed range as defined in the D1.1

The heating subsystem on the CED pilot line have been check

The Ultrasound and unfolding subsystems have been tested on the work temperature (70 °C)

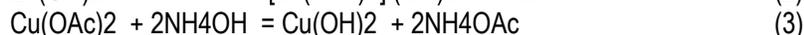
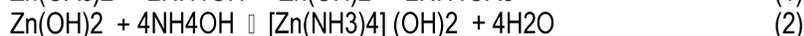
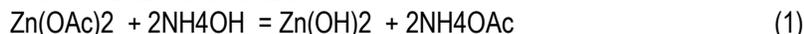
The washing and drying machine works at 1m/min textile speed.

SONO – coating chemical process and mechanism

The metal oxides nanoparticles were generated using an established working solution consisting of a metal ammonia complex in ethyl alcohol – water solution.

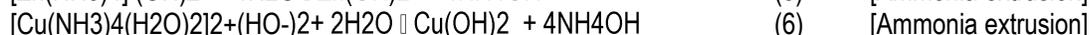
To prepare metal oxides (MeO) nanoparticles by means of ultrasound the starting reagents are: zinc acetate dihydrate: $Zn(OAc)_2 \cdot 2H_2O$ and copper acetate monohydrate: $Cu(OAc)_2 \cdot H_2O$ and ammonia solution (NH_4OH). Water – ethanol (1:9 v/v) mixture is used as solvent.

The chemical reactions are:



The metal ammonia complexes are water soluble. When ethanol is added the solubility of complex in water – ethanol solution is still high. (A translucent solution of zinc complex in water – ethanol is more accurate description).

When the ultrasonic transducers are on, the presence of an acoustic field generates cavitation bubbles. After several acoustic phases the bubbles collapse symmetrically in a homogenous media. According to the hot spot theory, the temperature and pressure developed locally could reach 5,000°K and up to 2,000 atmospheres. These extreme conditions decompose the zinc or copper complexes found in the surrounding layer of cavitation bubble by extruding the ammonia ligand from the complex structure leading, initially, to zinc or copper hydroxides while the collapsing bubbles are generating nano sized metal oxides:



Both hydroxides are insoluble in water or ethanol. Their lifetime is short and because of the high temperature at the end of bubbles collapse the hydroxides eject water leading to zinc or copper oxides in the form of nano particles (eq. 7 – 8) .

Final Publishable Report

$Zn(OH)_2 \rightarrow ZnO + H_2O$ (7) [Water extrusion]

$Cu(OH)_2 \rightarrow CuO + H_2O$ (8) [Water extrusion]

Due to the high ultrasonic irradiating surface of the transducers as well as the high ultrasonic power density, as soon as the nano particles are formed, they are fired into the fabric by asymmetrical bubble collapse, producing the nano particles impregnation effect (see below).

Asymmetric bubble collapse that fire nano particles into the fabric

Thus we conclude that the SONO – embedding process involves at least three steps:

- i. Metal complex decomposition to MeO NP's which are simultaneously fired into fabrics;
- ii. Not all of the MeO NP's formed reach the fabric and these remain suspended in working solution;
- iii. Some of the suspended MeO NP's follow the "throwing the stones" pathway and are embedded into fabrics; the rest remain suspended until the end of process.

Based on experiments performed at laboratory and pilot scale (on both pilot machines) the optimization of two pilot plant facilities for the production of textiles impregnated with nano-particles has been developed for those parameters which are vital for controlling the SONO coating process. The parameters were quantified to ensure a good coating of fabrics with the chosen NP's and to be useful for future scale up to industrial level.

The following parameters were found essential for SONO coating process and been optimized:

- Ultrasonic power density defined as w/cm^3 , and its value was established and optimized.
- The fabric distance to transducer is a fixed parameter in the machines as they are currently configured.
- Temperature plays an important role in the SONO coating process
- Fabric residence time (FRt), because this is a continuous process the fabric travels throughout machines with a certain speed in front of transducers. For any prospective industrial unit this parameter should be determined in real running conditions.
- Working solution flow rate is another important parameter that is strongly related to the SONO coating efficiency. The importance of this parameter is significant for the way in which the SONO process generates and impregnates NP's into fabrics. This is an important parameter determining the coating efficiency and its quality. Depending on the design of the next generation machine we propose to use fresh solution inside and maintain its chemical composition by replenishing it from outside solutions, in order to diminish the impact of oscillation of concentration due to the fabric impregnation. In this way the impregnation will be constant and the nanoparticles concentration on fabric will be uniform.

We should also emphasize that the sonochemical process is not having a 100% yield in transforming the metal ammonia complexes to metal oxides nanoparticles. In addition the sonochemical generated NP's are not 100% shoot into the fabric by the action of asymmetric cavitation bubble collapse. Therefore, at the end the working solution will have some metal ammonia complex, suspended nanoparticles and generated side products (ammonia acetate) which will accumulate if the working solution remains in the sonication tank and compensation is added as in the pilot units' long runs.

Depict potential mass balance for the entire SONO coating process

Mass balances for the SONO coating process cannot be calculated in the existing operational configuration. Taking into account the chemistry involved in this process one can see that ammonia is continuously regenerated (only a part that evaporated – lost, should be replaced) while ammonia acetate is accumulated. At this stage there are not reliable data for evaporation to quantify the ammonia necessary to maintain the right pH value and to replace that which is lost.

Mass balances for working solutions for both metals

The calculated compensation solutions for both metals are given in the picture below. It should be noted that the calculations were performed according to experimental work performed at laboratory level and does not take into account the "throwing the stones" embedding of textiles which involves already formed metal oxide nanoparticles.

Final Publishable Report

Mass balances for metal acetates compensation solution.

In the real run of the pilot machines the compensation solution contained 600 grams of zinc acetate to 10 L water. Having in mind that a part of zinc oxide nanoparticles are following the “throwing the stones” pathway, it is reasonable to consider that the long runs at pilot scale with 600 grams of zinc acetate is a good compensation recipe. Because a procedure for removing (continuously) the accumulated ammonia acetate has not been developed, it is impossible to predict a dynamic mass balance for the coating process (to offer solutions for lost materials i.e. evaporated alcohol and ammonia) as well as for removal of generated ammonia acetate).

Optimization of the textile products with simultaneous sonochemical/enzymatic/NPs coating of textiles

The objective of this work was the creation of active (anchoring) sites on textiles to improve adherence of the antimicrobial NPs. The fabric activation and antimicrobial functionalisation was meant to be carried out via simultaneous enzymatic/US/NPs treatment of cotton fabrics. This process is expected to improve the uniformity of NPs distribution on the surface of the textile material and to enhance the stability of the NPs coating during exploitation of the fabrics in medical environment.

Cotton fabrics were ZnO coated at lab scale using simultaneous cellulase/US/ZnO NPs process. A protocol for improved NPs coating of textiles using enzymes and US is currently being designed. The following evidences support the feasibility of the simultaneous process:

- Cellulase pre-treatment enhances the sonochemical deposition, adhesion and uniformity of ZnO NPs onto textiles.
- Cellulase activity is not affected by the presence of ZnO NPs.
- ZnO NPs are stable in aqueous medium where the process should be carried out, allowing for their simultaneous application with US and neutral cellulase.
- Application of US together with cellulase enhances the hydrolytic efficiency of the enzyme.

Major achievement:

The enzymatic pre-treatment provided:

- a. smaller size NPs with higher activity at lower concentrations
- b. increased homogeneity/distribution of the sonochemically deposited ZnO NPs on the fabric surface
- c. improved coating durability

Simultaneous hybrid (biopolymer-NPs) antimicrobial embedment using US

This approach is about the enhancement of the antibacterial properties of sonochemically-coated fabrics using a simultaneous application of intrinsically antimicrobial chitosan and inorganic NPs, e.g. ZnO. Since both CS and ZnO NPs are efficient bactericide agents, the preparation of hybrid coating including CS and ZnO NPs was expected to produce antibacterial materials with excellent antimicrobial performance. Hybrid coatings composed of CS and ZnO were thus deposited sonochemically on cotton fabrics and their antimicrobial properties were further evaluated.

Major achievements:

- a. Efficient and non-toxic antibacterial textiles were produced via a one-step simultaneous sonochemical deposition of ZnO NPs and chitosan.
- b. US-assisted embedment of hybrid chitosan-ZnO NPs was optimised in terms of antimicrobial reagents concentration, pH and processing time towards obtaining highly efficient antibacterial textiles.
- c. Hybrid chitosan/ZnO coating displayed improved antibacterial properties when compared to the coatings comprising the individual components (ZnO or CS).
- d. Deposition of chitosan substantially improved the durability of the antimicrobial effect of ZnO coatings to withstand up to 10 washing cycles typically applied in hospitals
- e. Chitosan within the hybrid coating improved their biocompatibility.

Product characterization and end-user validation (BIU)

Final Publishable Report

WP6 is led by BIU, and deals with the Product characterization and end-user validation. The partners involved in the current WP are: CU, AITEX, INCDTP, EMI, PR, UPC and DAVO.

3 Main categories of coated fabrics were produced during the project:

- Fabrics from lab - device located at BIU : M1-M18
 - set the characterization parameters – practice
 - simultaneous coating (biopolymers, enzyme + Metal Oxides)
- CuO coated fabrics by 2 new SONO pilots : M18-M36
 - 2 modes of coating: “in-situ” and TS
 - Comparison between the performance of 2 lines
- ZnO coated fabrics for trials at EMI : M36-M48

During the first 18M of the SONO project characterization parameters were collected. These parameters include definitions of what should be included in the characterization tests and what are the target numbers we would like to reach for the various parameters (Table below). Since the first prototypes developed in the SONO project were established only after 18 months of the project, the partners involved in the characterization have practiced on fabrics coated by BIU's small pilot machine during the first 18M.

The partners have carried out the following tests:

BIU has coated various types of fabrics with ZnO and CuO nanoparticles. The fabrics are: 100% cotton, 100% cotton treated with enzyme and chitosan by UPC, colored and combined fabrics with and without UPC treatment. BIU has prepared 50meter of each type and send it to CU, AITEX and INCDTP for further investigations. To each sample sent to the partner a number was assigned from the “sheet” provided by PR.

The antibacterial activity of cotton fabric coated with ZnO NP's and of fabrics that were pre-treated with enzyme/chitosan and then coated with ZnO NP's is presented in the following graphs. Both the enzyme treatment stage, and the added chitosan coating, appeared to provide an improvement in the antibacterial effect in comparison to the original ZnO NP coating. The Chitosan coating appeared to provide the most significant improvement.

During the first year of the project the antibacterial testing focused on ZnO fabrics prepared at the lab scale. Some of the results from this testing are shown in Figure 6.1. Although ZnO cotton fabrics showed activity against all of the test bacteria the levels were not very high against some of the Gram negative species. The antibacterial activity after 10 wash cycles was also not very good in some cases. Enzymatic pre-treatment of the cotton with cellulase improved both the antibacterial activity levels and the wash durability. This enzymatic treatment resulted in the formation of smaller NPs on the fabric fibres increasing their antibacterial activity 2. The addition of the antibacterial biopolymer chitosan alongside ZnO NPs also improved antibacterial activity levels and wash durability 3.

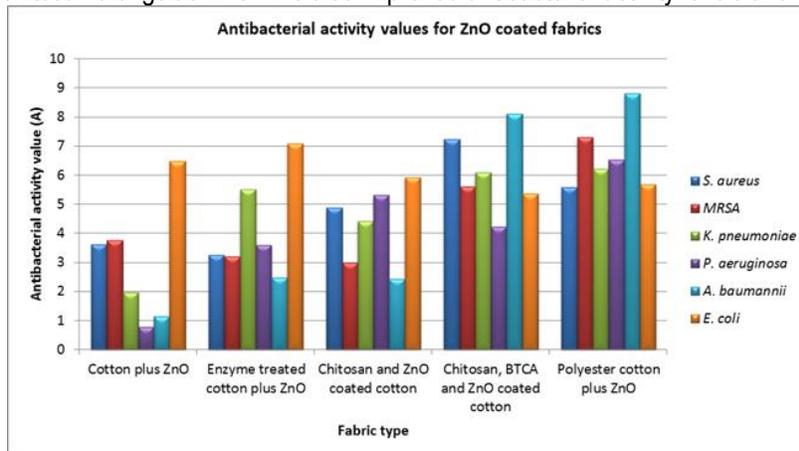


Figure 6.1 Antibacterial activity of ZnO fabrics in combination with enzyme pre-treatment of cotton, chitosan and BTCA. The testing was carried out according to the absorption method from ISO 20743.

Final Publishable Report

Antibacterial activity values of $A > 1$ were taken as an indication of significant activity. $A > 3$ indicated strong activity.

Inclusion of the covalent cross-linking agent butanetetracarboxylic acid (BTCA) with chitosan and ZnO NPs resulted in high levels of antibacterial activity against all of the test bacteria and improved wash durability. However, the work with BTCA was not continued due to negative effects on the physical properties of the fabrics. As shown in Figure 6.3.2, polyester cotton (PEC) mix fabrics coated with ZnO NPs displayed excellent levels of antibacterial activity.

Silver and triclosan coated PEC fabrics were tested for comparison with the SONO fabrics. They also showed very high levels of antibacterial activity against all of the test bacteria but displayed poor wash durability.

In the period M19-M36 there has been significant progress both in technical (maintenance) and scientific features of the sample characterization database (Sample DataStore or DS). In order to improve response times of the web site, PR renegotiated contracts with internet service providers and obtained 50% more upstream (from server to clients) bandwidth on machines serving SONO project.

According to Partners' needs, PR has developed reporting facilities of the DataStore. In particular, it has been integrated sample history browser and file browser, so essential properties of samples can be seen together with raw data, e.g. TEM images, as presented on the picture below.

Partners have contributed with definition of textile characterization parameters. In the reporting period it has been added over 100 new results describing textile samples, antibacterial properties and sonochemical processes.

Main conclusion from the first 18M is as follows :

- The particle's size of ZnO / CuO coated on cellulase treated fabric is smaller than on surface of the untreated one (Milestone 09).
- Improvement of antibacterial activity with chitosan + ZnO on cotton (Milestone 09).
- CuO and ZnO on PEC active against all test bacteria.
- Antibacterial activity of CuO PEC found to be durable for up to 100 wash cycles. Activity and durability of ZnO and CuO better on PEC than on cotton (Milestone 06).

During the second period of the project (M19-M36) there was a move from the testing of samples prepared using the lab scale system (at BIU) to the testing of samples prepared using the two pilot machines; the magnetostrictive system made by VIA and installed at DAVO, and the piezoelectric system made by CED and installed at Klopman. For the pilot scale machine experiments, CuO continued to be the focus, partly, because it changes the colour of the fabrics, aiding assessment of the coating quality. The second reason for using CuO is that in addition to the sonochemical coating process, where copper acetate in ethanol and water is converted in situ to CuO, a "throwing the stones" method can be used. In the "throwing stones" method the fabrics were ultrasonically coated from an aqueous suspension of CuO nanoparticles (commercially sourced CuO). This mode enables to proceed at the pilot scale without the use of large volumes of ethanol. In addition, identical experiments were carried out on both pilot lines, at DAVO and KLO. The same reaction conditions and a similar type of fabrics were used for the identical experiments.

Figure 6.2 presents an image of fabrics from the 2 lines, coated in the throwing stones mode using commercial CuO NP's (grey) (from an identical source), and in the "in-situ" mode using Cu(Ac)₂ – brown. Even by the naked eye observation it can be seen that the fabrics coated by the KLOPMAN machine have a uniform coating. In contrast, the fabrics coated by DAVO's machine, have a non-full coverage, and some "white" regions are observed. A deeper observation of coating was done by using the Scanning Electron Microscopy (HRSEM) and will be presented below.

Final Publishable Report



Figure 6.2 Image of coated fabrics from 2 lines

From the characterization results received in P2, the "in-situ" mode of coating provides a more homogeneous coating than the Throwing Stones mode. Machine located at KLO produces a better coating in terms of amount and homogeneity than DAVO's machine. The coating is retained on the surface even after 60 cycles of washing and only a small amount of ions are leached from the surface during the initial washing cycles.

Cotton impregnated with CuO NPs showed good levels of antibacterial activity against each of the test bacteria and also good wash durability⁴. A significant milestone in the project was the demonstration that PEC fabrics impregnated with CuO retained antibacterial activity even after 100 wash cycles at 75°C. In Figure 6.4 it can be seen that antibacterial activity levels after washing only dropped below significant levels ($A = 1$) in the case of *Pseudomonas aeruginosa*. When CuO was combined with chitosan significant antibacterial activity was retained against all of the test bacteria even after the 100 wash cycles.

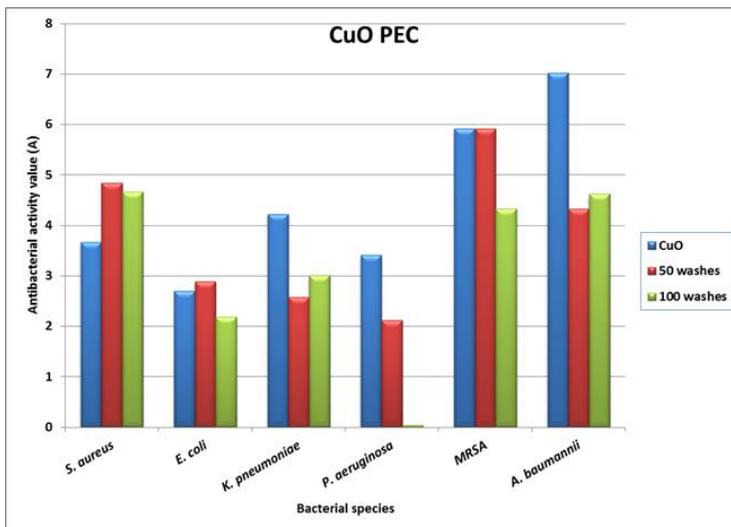


Figure 6. 4 Antibacterial activity of CuO PEC fabrics before and after washing. The 50 and 100 wash cycles were carried out according to ISO 63305 at 75°C and with a neutral detergent (ECOS).

Final Publishable Report

Antibacterial testing of SONO fabrics from the pilot scale machines commenced during the second year of the project. Some of the results from these tests are presented in Figure 6.5a and Figure 6.5b.

The antibacterial activity of the standard CuO NP 'in situ' coatings was found to be better than the activity of the CuO TTS coatings. This was most evident with the Gram negative species (*E. coli*, *P. aeruginosa* and *A. baumannii*). Scanning electron microscope images of the fabrics revealed that the SONO process produced a homogeneous layer of CuO NPs on the fibres. The CuO deposited via the TTS process, however, was unevenly distributed and present as micrometre scale aggregates. Reducing particle size has been shown to increase antibacterial activity.

The antibacterial activity levels of the SONO fabrics prepared using the two different machines were very similar. The fabrics produced using the piezoelectric machine at KIOPMAN was slightly better in terms of wash durability. Antibacterial activity was retained after 60 wash cycles at 75°C. This may have been due to a higher starting amount of CuO on the Klopman fabrics (0.9 % w/w CuO) compared to the Davo fabrics (0.37 % w/w CuO). The TTS coatings were also slightly better from the Klopman machine with improved antibacterial activity against the *P. aeruginosa* and *A. baumannii*.

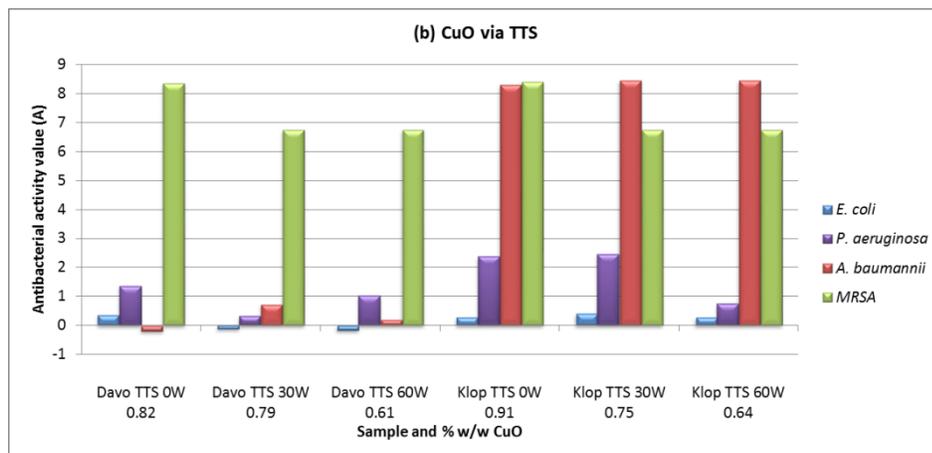


Figure 6.6 antibacterial activities before and after washing for the CuO PEC fabrics produced using the pilot-scale machines. Upper graph (a) CuO NPs formed and deposited in situ using the SONO process. Lower graph (b) CuO NPs added from aqueous suspension via the ultrasonic 'TTS' technique. The 30 and 60 wash cycles were carried out according to ISO 63305 at 75°C with a neutral detergent (ECOS).

In conclusion, the ZnO and CuO SONO fabrics displayed excellent levels of antibacterial activity against a range of bacteria associated with hospital acquired infections. The SONO PEC fabrics also displayed excellent wash durability with retention of antibacterial activity after 100 hot wash cycles. The SONO fabrics prepared using the pilot-scale machines were as good as, if not better than, those produced using the laboratory scale SONO process.

One of the major milestones of Period 3 (M36-M48) was the production of ZnO coated fabrics for the hospital trials at EMI in Bulgaria. The reason for using zinc oxide is that, the ZnO is white in nature and after it's coating the color of the substrate does not change. The latest is a very important parameter for medical clothes - to keep the original color of the textiles, namely white. The type of textiles that were used for the clinical trials was woven made from 100% cotton treated sonochemically with ZnO nanoparticles by in situ synthesis method. The study was conducted at the University Multiprofile Hospital for Active Treatment and Emergency Medicine "N.I. Pirogov" (EMI) and included a total on 37 subjects (21 active and 16 controls).

Klopman and DAVO have produced approx. 2000 meter of ZnO coated cotton. 1500m was produced by KLO and 500m by DAVO. The coating was done in a continuous mode by adding a compensation solution every 10 min. The results clearly indicate that the fibers are coated with a constant amount of ZnO (~0.3%wt). The particles distribute along the fibers and the results are actually the same for both machines.

Final Publishable Report

At the net stage the ZnO coated materials were used for confecting medical staffs for clinical trials. The confection was done by DAVO.

The antibacterial properties of ZnO coated textiles ("in-vitro" tests) that were produced in a continuous mode were revealed a high level of activity against all tested bacteria.

In addition, during the last year of the project the coated fabrics were tested against the bacteria that are resistant to antibiotics. The sonochemically coated textiles were found to be active against all 6 multi drug resistant bacteria.

And finally, it was shown that the sonochemical textile maintains lower level of microbial growth in comparison with the standard hospital textiles used in the control group. Very important finding is its ability to maintain relatively constant and low level of growth of one of the most difficult-to-treat nosocomial pathogens – the MDR *Acinetobacter baumannii*. These strains are ubiquitous and used to survive in unfavourable conditions for long time. There are very few antimicrobial agents or disinfectants that are still effective to eradicate them.

Economic and environmental studies (EP)

Since the early stages of the project development WP7 activities contributed to the definition of processing criteria in order to allow comparing different solutions based on sonochemical finishing. Experiments were designed in order to run same processes on the two different pilot lines; several processes were investigated. Data inventory for Life Cycle Costing (LCC) and Life cycle inventory (LCA) were performed, including processing parameters and measurements as well as inventory of units data to support LCC. First LCC effort was devoted to the units systems and subsystems analysis, accompanied by a functional and costing analysis in order to identify the higher impacts in development phase. The WP7 investigation was then focused on processes. On the base of LCA and LCC obtained data a bivariate analysis allowed to compare processes routes by fixing the processing unit and, as well as to compare the results of same processes referred to the two pilot machines. This bivariate approach provided also useful information on environmental sustainability of magnetostrictive vs. piezoelectric technology and allowed identifying the most effective processing routes to attain antibacterial functionality with minimum environmental impact and for enhancing cost effectiveness. Indeed comparative data interpretation phase provided indications already at development stage. The LCC methodology has been applied to the analysis of the economical sustainability of several processes executed on both industrial pilot scale SONO systems based on magnetostrictive (MG) and piezoresistive (PZ) Ultrasound transducers. This LCC assessment had the following principal objectives based on multivariate analysis:

- to compare two type of functionalisation process methods: 'throwing stones' (TS) and 'in situ' (IS) generation of active nano-particles (NPs)
- to compare the same process economic efficiency run with the two units (MG and PZ)
- to evaluate processing and processing units economic performance including geographic peculiarities such as reagents cost, labour cost, etc.
- to compare obtained data with the current benchmarks

The selected cases to be analysed are represented in the following diagram:

Final Publishable Report

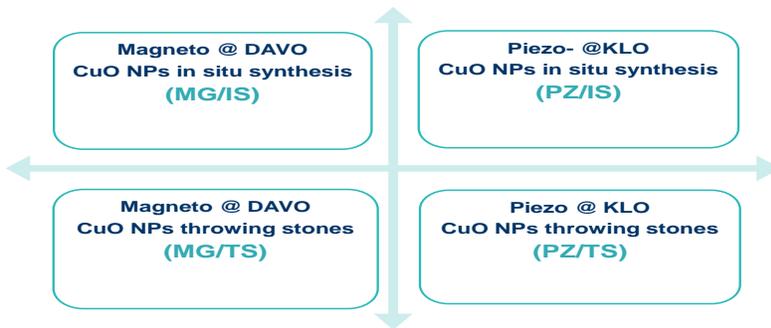


Figure 7.1 Schematics of the analysed cases for processes and technology comparisons

The different cases were investigated on the basis of the identified technical unit, corresponding to one kilogram of treated fabric, as a function of different process parameters, such as power input.

The comparative main results shown that the in situ synthesis of CuO with Magneto resistive unit (MS/IS) is the process providing the most economical sustainable solution, when the lowest power input is set; and that the cost of magneto resistive in-situ processes with Copper acetate cost are mainly affected by RTD and unit depreciation for the first exploitation period. Results shown that after the RTD and initial investment depreciation period, the process cost is already competitive with the solutions present on the market today. It was observed that enhanced throughput allows to drastically abating cost sources for depreciation of RTD and unit by rendering the solution totally compatible in term of cost. However considering process parameters, the power input increase of three times induces a proportional cost increase of other cost sources.

Further to technical unit comparisons, the system boundaries and the functional unit of the analysis have been characterised according to the need of defining invariant quantities that have also technical meaning for comparing different processes or treatment of different substrates (e.g. 100% cotton and cotton/PET blends, different process recipes, etc.). Data analysed were derived by inventories on specific classes of processes whose data were provided by technical partners.

Specifically, the key items identified with LCA determined that PZ system in both different process SONO is characterized by a lower Primary Energy Demand vs. the; MG pilot and consequently by a lower GWP100yrs, as expected.

Reviewing the other parameters, the Ozone Depletion Potential is negligible for all the scenarios considered and potential impact to water discharges (Acidification Potential and Eutrophication potential) are minor and comparable for both pilot systems.

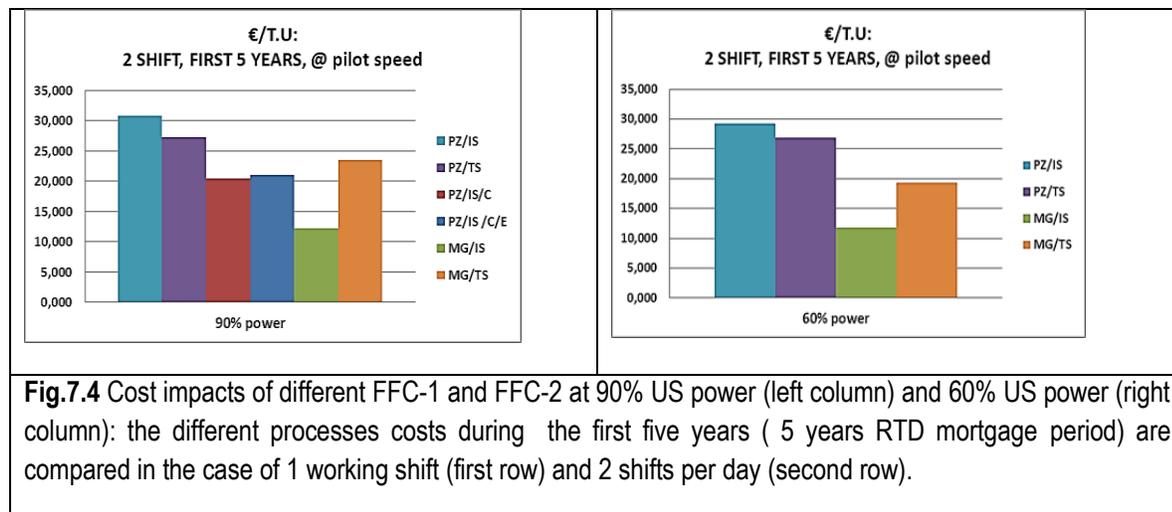
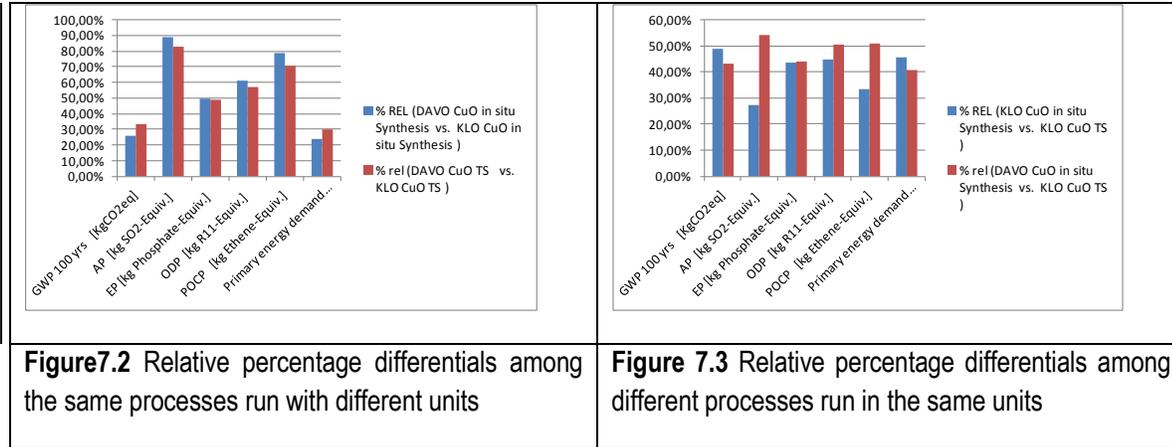
When comparing processes it was quite striking the difference in impact due to the in situ synthesis with respect to the throwing stones. This behaviour is confirmed on both pilot units and for all the environmental impact parameters for which relative increases range between 30% and 50%.

While the comparison of the same processes run in the two different pilot lines provided an explicit relative difference between the two units for both processes: for all the impact parameters the VIA pilot shown a relative increase ranging from 30% up to 90%.

From the LCA analysis it was possible to see that the less impacting process is the throwing stones (TS/IS) run on CED pilot line.

For what pertaining cost issues, it has been pointed out that the power input has a moderate impact on final cost.

Final Publishable Report



Towards the last phase of the project the optimized processes were compared with industrial benchmark with reference to the selected technical and functional unit in order to provide indications towards industrial scale up of product and process cost effectiveness as well as qualification and quantification of the process environmental impact. Evaluation of the impacts of the new products on medical textiles regulations and market assessment to specifically evaluate market receptivity and target was performed to better support future exploitation actions.

Cost effectiveness was based on comparative cost assessment of the optimized SONO Pilot processes referring both to the technical and to the functional unit. Comparisons were obtained among the solutions investigated within SONO project and comparisons between the reference SONO process and the industrial selected benchmark was possible. Analysis and assessment has been performed for the addressed processes obtained with up scaled units installed at DAVO and at KLOPMAN. In the LCC several scenarios have been considered. The overall LCC was also provided in comparison to LCC results for industrial benchmark process. The analysis included:

- a. Process Planning and RTD efforts directly connected to the process development (RTD),
- b. Investment cost, included as amortization / depreciation cost
- c. Running cost,

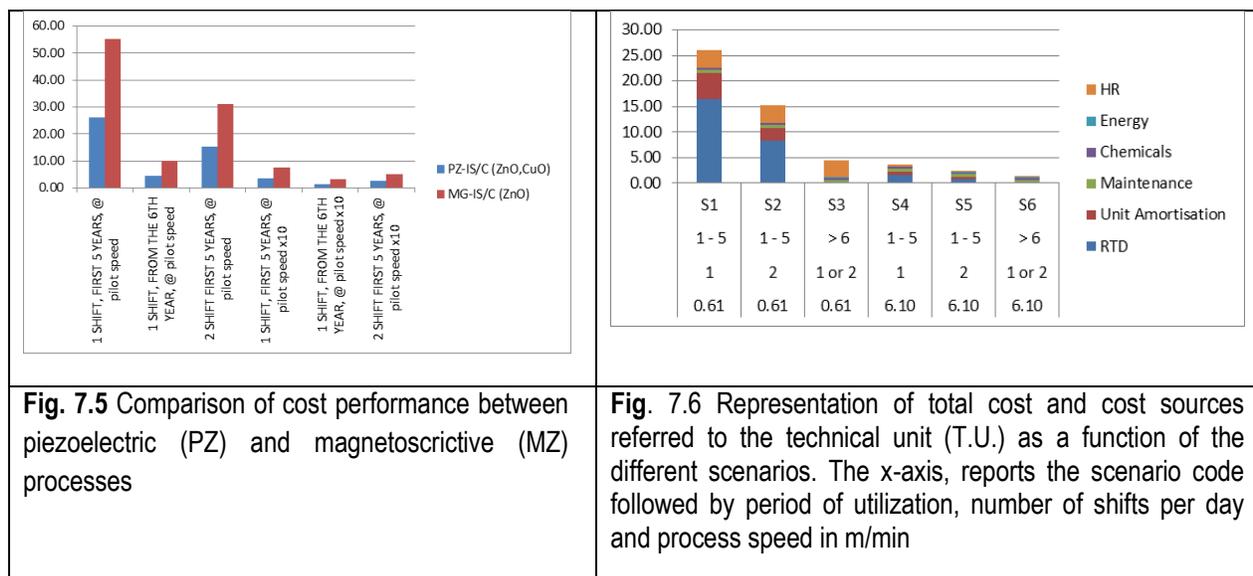
and has been carried out considering different scenarios applied to both processes and to both units:

- i. five years running of the functionalization unit
- ii. after the first five years operation (sixth year is taken as example)
- iii. production with one daily shift of workers
- iv. production with two daily shifts of workers

Final Publishable Report

- v. production through put as per pilot unit set production speed
- vi. enhanced production throughput, by increasing the speed of ten times (x10) in order to envision results on industrial throughput compliance.

The comparative cost analysis within SONO processes allowed identifying the piezoelectric based process for in situ synthesis of metal oxide nanoparticles as the best cost effective one in all considered scenarios with cost savings amounting to 50% of the magnetostrictive based process, as detailed in the following diagram.



In order to assess the sustainability of the investigated processes, a detailed analysis on the major cost sources was performed. This will also provide further guidance at industrial implementation stage in order to enhance process efficiency and cost effectiveness. The obtained results prove that the economic sustainability is achieved for increased process speed or when depreciation and RTD costs are not influent on process cost anymore. In this framework the most critical cost items are depreciation of RTD effort in the first five years operation and human resources as fixed cost. In order to face this major cost, two strategies are proposed: production intensity enhancement (increase the number of shifts by compressing RTD costs), production capacity (by increasing the processing speed). Evaluations of both scenarios have been considered.

The interesting result is that, when comparing these scenarios with an industrial assessed process (the benchmark), already at pilot pre-industrial stage the economic competitiveness of SONO solutions is achieved. Indeed, when considering the renormalization of cost based on the functional unit defined for Q60 (quality factor at 60 washing cycles), the comparative cost impact is dramatically reduced and the scenario S3 (i.e. process at 0.61m/m from the 6th year operation) provides a cost which is only 88% of the benchmark cost (duly renormalized on F.U.): this means that the PZ-IS/C (ZnO) process F.U. provides already economical sustainability when the process is run at 0.61% of the industrial benchmark process speed. This result is based on experimental data and on antibacterial activity-AA level tested samples, which allow measuring the process functional output performance. Indeed the scenario S3 (6th year operation scenario at 0.61m/min) is the most reasonable one since it represent the most comparable condition with respect to benchmark and is based on experimental direct data. This result provides the measure of the SONO process competitive advantage. By increasing process speed the cost effectiveness further increases

Referring to the environmental sustainability, LCA results, firstly, were computed on the basis of the selected technical unit and then renormalized on the functional unit. Analysis and assessment has been performed for the addressed processes obtained with up scaled processes run on the sonochemical units installed at DAVO's (based on magnetostrictive technology) and KLO's (based on piezoelectric technology). The LCA was also applied to qualify the selected industrial benchmark process in order to provide comparisons between SONO solution and industrial processes in use. The specific sonochemical processes analyzed are:

- ZnO and CuO in situ synthesis processes with compensation run on Piezoelectric pilot unit

Final Publishable Report

- ZnO in situ synthesis process with compensation solution run on the Magnetostrictive pilot unit
- Standard industrial benchmark process based on silver salts currently employed at KLOPMAN's for performing textile antibacterial finishing

The antibacterial activity level allowed defining the quality factor to re-normalize the LCA (and LCC) results. This allowed to express results obtained for the technical unit with respect to the functional unit, providing a more general and representative comparative basis.

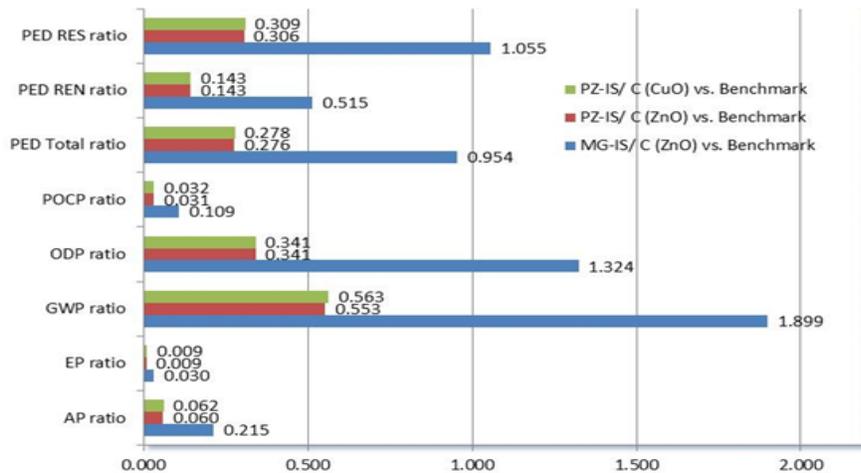


Figure 7.7 Relative impact assessment of piezoelectric process for in situ- synthesis of Copper oxide, Zinc oxide with respect to benchmark silver based process. Relative data are referred to the T.U.

The environmental performance of the investigated processes has been quantified with the standard impact parameters and energy demand in order to compare the piezoelectric based processes and the magnetostrictive based processes. Results show that the piezoelectric unit exploits an antibacterial finishing process which requires less direct energy, thus determining lower environmental impact. Impact is not sensitive on the metal oxide synthesized (the chemistry does not sensibly change the output impact parameters), whereas the magnetostrictive process, when compared to the benchmark in some cases exceeds the benchmark values. This is valid for the analysis based on technical unit, which is a more conservative comparative basis. When renormalizing on the functional unit, that is on the product antibacterial functionality durability, all sonochemical processes show lower environmental impact.

The impact of new products on medical textile regulations have been assessed first by identifying the application context and the reference European regulations. The main result of this evaluation is that the developed products in SONO project could be placed in the European Market if the active substances are included in the ANNEX II of 1451/2007, as products Type 2; according to new regulation No 528/2012 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 22 May 2012 concerning the making available on the market and use of biocidal products. So the manufacturer of the chemical products or the manufacturer of the antibacterial textile must apply for the evaluation of them in order that they can be included in this ANNEX, as product type 2. This result is quite important because identifies a market segment in which the SONO products may be placed avoiding lengthy and risky assessment to comply with medical device directive (MDD). The compliance with less stringent regulations will allow SONO product to diffuse in the market and this does not hamper the possibility of being accepted also within the MDD in a second time. The impact of SONO products performance on regulations may also be relevant considering the level of antibacterial activity and durability of the same, which is far beyond the products in use.

What considered above has also relevant implications on the market assessment. The actual competing products are based on silver salts and applied to textile products that have not a market potential comparable to that targeted by SONO products. One of the main problems in hospitals with regard to these products is nosocomial infections because of the contact between the patient and infected bed sheets and dress gowns. SONO antibacterial treatment addressed this specific market need. Forecasts on the application of SONO technology report that after this

Final Publishable Report

technology will be fully developed into an industry the added cost of having the fabric coated will not significantly affect the final product price, a price that should remain very competitive and offer a unique functionality that can comply with the infection prevention program of any health system for the safety of patients and for the system cost effectiveness. The antibacterial fabric realized with sonochemical process brings an intrinsic added value to the non-surgical textile products that have practically no geographical limitation, thus leveraging the SONO products marketability potential to achieve saturation of global demand for nosocomial applications.

Design, modeling and evaluation of a scaled up (x5) industrial production (CED)

The full chemical process from SONO project for an antibacterial coating includes four tanks (see scheme):

- A reagent tank
- A Sonochemical tank
- An end squeezing roller waste tank
- An Ethanol recovery tank

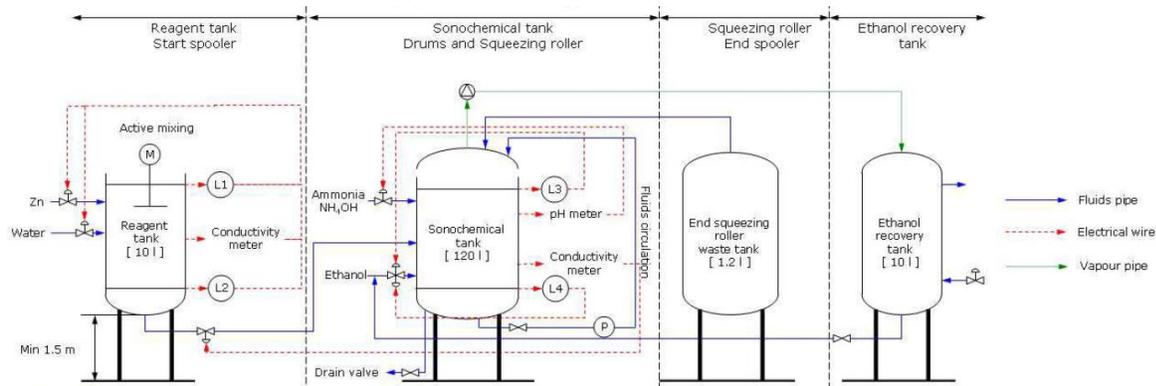


Figure 8.1 : The chemical process scheme of the machine

CED has provided KLO with both the Sonochemical Tank and the End Squeezing roller waste tank, together named as the Sono Tank.

CED machine unfolds the textile from a start spooler, pass and treat it through the sonochemical tank, squeeze and fold it in the end spooler before the washing tank machine (see scheme).

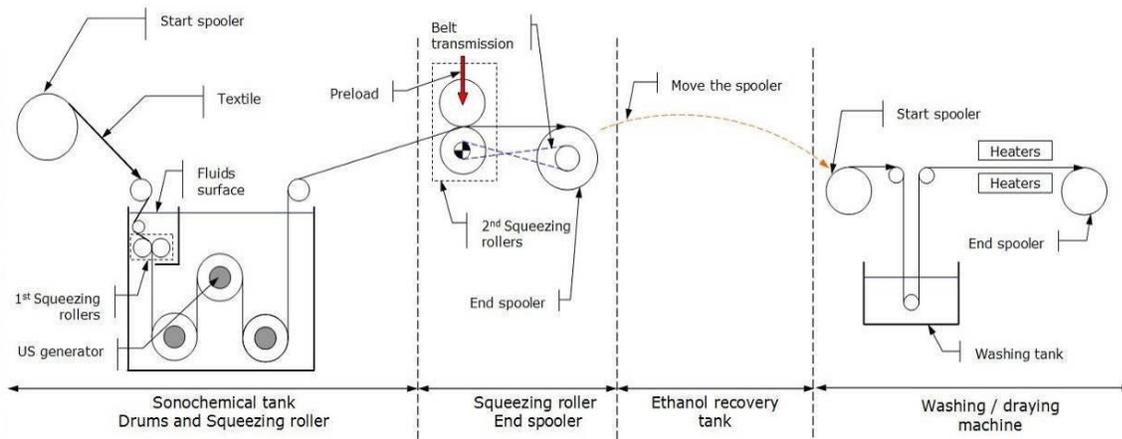


Figure 8.2 : The textile unfolding representation scheme

The sonochemical tank comprises:

- the metal tank

Final Publishable Report

- the squeezing rollers
- the driver rollers
- the Ultra Sonic generators (transducers)
- the Ultra sonic electronic drivers

The upscale sonochemical tank target from the CED side are :

- to treat width textile $w=1.5$ m
- at speed $s=10$ m/min

The present experience at CED as regards this objective is the following:

First tests of CED sonochemical tank have shown the feasibility of the sonochemical coating process. But there are limitations:

- The width is max 0.5m;
- The max speed is unknown; Present tested speed giving good coating is below 1m/min.

Under our request, Pr Aharon Gedanken BIU has asked KLO to make tests with speed up to 10m/min. Today the results of the tests from KLO are unknown.

Limitation in width comes from 2 width limits :

- LW1 - the widths of sonochemical tank and roller
- LW2 - the length of the Ultra Sonic generators (transducers) and their power supply.

Limitation in speed comes from 4 speed limits:

- LS1 - the speeds of the mechanisms (for ex roller motors) which is responsible for the fabrics motion
- LS2 - the speeds of the control which is responsible for the control of the fabrics motion
- LS3 - the sono chemical reaction responsible from the coating on the fabrics
- LS4 - the acoustic power at the fabric which accelerates the sonochemical reaction

The width limits are analysed as following:

LW1: the internal width of tank mechanics is a bit larger than 0.5 m. There is no difficulty to increase this with. Just the fact the internal width of 1.5 m leads to a large tank which is no easy to handle and transport.

LW2: the length of the active part of the tubular transducers is ~ 0.75 m.

Keeping the same technology of transducers, it is not possible to reach 1.5 m width. The 0.75 m tube length is a maximum. With longer tube the transducer efficiency is not homogeneous and the power is decreased. There is already not perfect uniformity in the deposit with this kind of transducer. Simulations results show this phenomenon.

Tests have been done on specific tubular transducer designed by CED using our lab facilities like laser interferometers:

Even if these transducers are working as expected, their limitation in length is a problem for an up-scale process. For this reason CED has developed new Modular Ultrasonic Transducers (MUT). MUT are 0.75m long like the tubular transducers. However unlike the tubular transducers; several MUT can be aligned to cover large widths keeping high power and good homogeneity. For example 2 transducers can cover 1.5 m width, 4 transducers can cover 3 m width, etc.

Final Publishable Report

This modularity is the first advantage of the MUT. However this modularity has not been experimented yet, because the tank doesn't allow it. The risk is in the acoustic interaction. Such a risk can be reduced by acoustic modelling and by playing with some dimensional parameters. This may impact as a partial redesign present of MUT.

Traditional tubular transducer can't be coupled to each other easily and the acoustic pressure generated is decreasing along the tube. This phenomenon leads to a non uniform deposit on the textile for the SONO application. Thanks to the innovative design of the modular transducer, it is possible to stack as many transducers as requested and also to obtain a better uniformity in the acoustic pressure generated along each transducer.

In order to validate the efficiency of this kind of modular transducer, a first prototype has been made with just one module. The results are very close to the last expectations and we are optimistic about this new solution which provides similar performances than the traditional transducer but with a larger surface and a modularity feature.

The new version of CED sonochemical reactor to accommodate fabric of width 1m (12) is based on the additional transducer on the sonochemical tank. In order to accommodate the 1m wide fabric, the width of prototype reactor was extended to 1.65m. At the one end (front end), three transducers (each connected to a tubular resonator) were installed in a position identical to that in the prototype reactor. At the other end (back end), three additional transducers positioned as in the former three were employed. However, the length of the resonator connected to each of them mirrored those used in the front end (see here after).

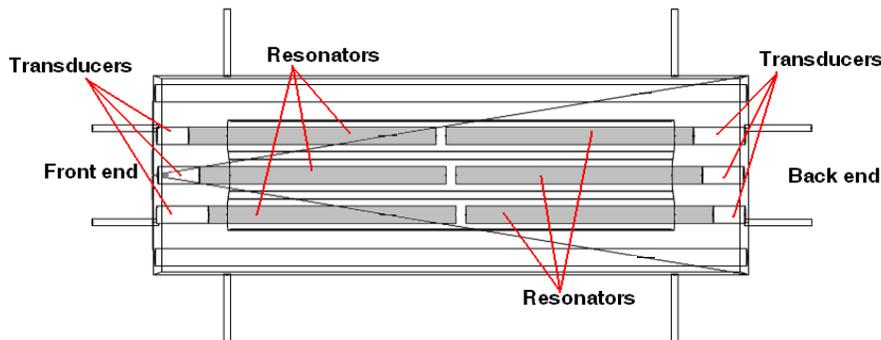


Figure 8.3: The CED tank scale up proposition

Several solutions are proposed in order to overcome the problems encountered on the pilot lines. The new designs are presented in the following illustration

Final Publishable Report

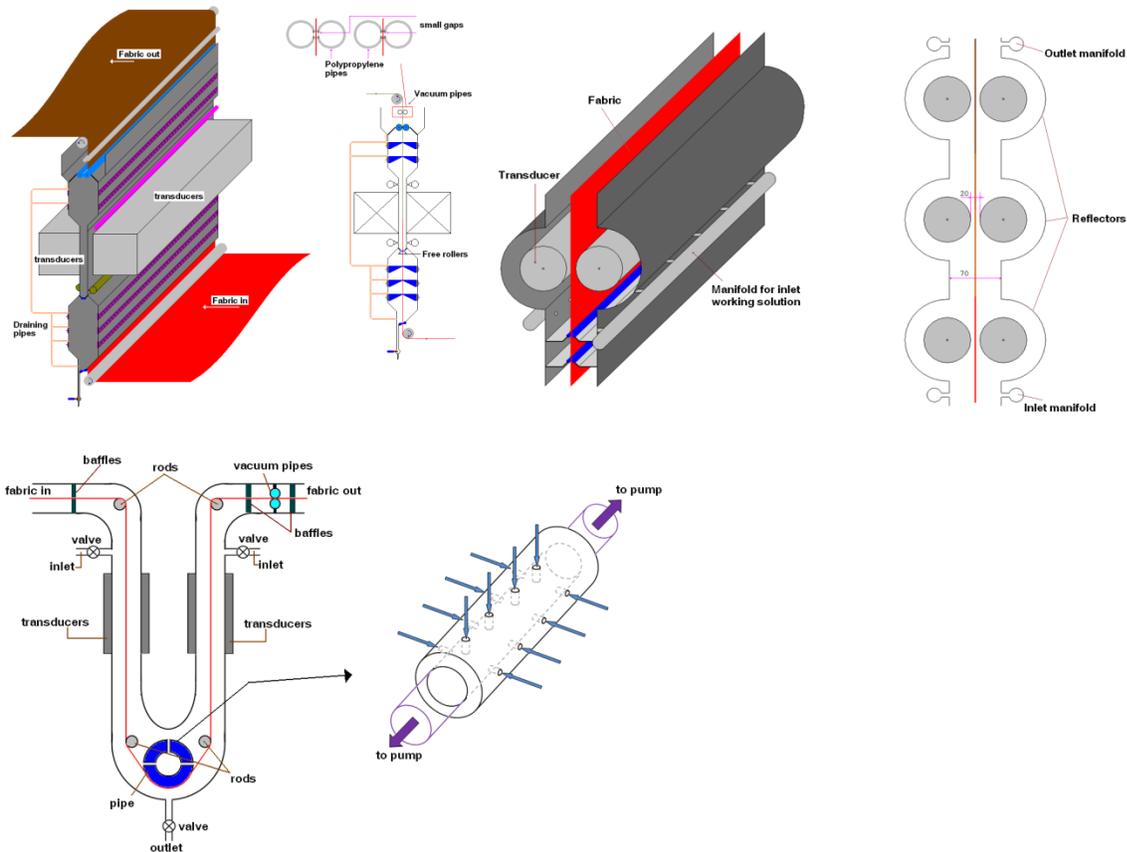


Figure 8.4: The tank modification proposed based on simulation feedback

The significant results obtained in this work package are

- The up scaling process has been provided in the aim to design an industrial machine based on the feedback of the pilot lines.
- A better understanding of the chemical process show that one tank (sonochemical tank) is required compare to the pilots lines (no need of reagent tank).
- The ultrasonic transducer has been optimized in the aim to fit with coating application instead of the ultrasonic transducer used in the pilot's lines which are a commercial one.
- The process cost per kilogram of coated textile is evaluated for different cases during and after the amortization.
- The process cost drop from 4.45 to 1.4 €/kg by increasing the speed by ten from 0.6 to 6 m/min.