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**ULTRATEC**

Project title:
**Acceleration of Textile Processes by Ultrasound Technology**

Instrument: **Specific Targeted Research Project (STREP)**

Thematic Priority: **Priority 3 - NMP**

# PUBLISHABLE FINAL ACTIVITY REPORT

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Project coordinator name: **Marco Barbieri**
Project coordinator organisation name: **Tecnotessile (TTX)** Revision: **0**

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## 1. Project execution

The general project objective was supporting long-term innovation of the European textile industry and increasing its competitiveness by means of the generation of relevant scientific knowledge to develop novel ultrasound (US) processes for textile and nonwoven applications.
The expected end-results of the project were the following:

- generation of relevant scientific knowledge in sonomechanics, to be used to enhance mass transfer in continuously operated textile wet processes;
- generation of new knowledge on sonochemistry applied to textile processes, thus obtaining: synergisms between acoustic fields and enzymes in homogenous and heterogeneous systems, novel coating processes, and novel processes in the nonwoven production;
- development of dedicated design rules for ultrasonically driven processes in the textile industry;
- development of dedicated design rules for transducers and ultrasonic equipment for different textile processes.

All these results were achieved by the partners; in the next paragraphs, more details about these results are reported.
The project outcomes allow to the knowledge-based application of the ultrasound technology in several textile processes. This is a major breakthrough compared to the state-of-the-art. In fact, so far a so-called "black-box" approach has been commonly used when studying applications of ultrasound. The black-box approach consists in the performance evaluation of a system as a whole, i.e. black box evaluations consider the overall performance of a system without reference to any internal components or behaviours. The application of the black box approach when studying ultrasound applied to textile processes has resulted in inefficient processes giving contradictory results, since in ultrasound most parameters and variables are not independent of each other. This has prevented an industrial-scale application of the ultrasound technology in many textile processes where the ultrasound technology would have the potential to provide several benefits. On the contrary, as reported in the next paragraphs, a fundamental understanding of the ultrasound technology applied to different textile processes was achieved by the project partners. The gained knowledge will function as starting point towards the industrial application of fast, ultrasound-based processes in the European textile sector. Hence, in medium term the project will have a major impact in the improvement of the competitiveness and sustainability of the European textile industry, because the industrial-scale application of US in the processes investigated by the partners will result in the following benefits: a) reduction of processing time, allowing shorter production runs; b) savings of energy and chemicals, with the consequent economical benefits for the industry due to the reduction of costs, and environmental benefits, due to the reduction of discharges of hazardous substances; c) enhancement of biotechnical/enzymatic processes in textile manufacturing, thus contributing to the diffusion of new eco-efficient technologies based on biotechnology in the textile sector.
The work programme was carried out by a consortium representing the state of the art in term of complementarity for the ultrasound and textile areas, coordinated by the Italian textile research

centre Tecnotessile (www.tecnotex.it; tel. +39-0574-634040). The basic research in the field of ultrasound was generated by the University of Twente (UT-TXT, the Netherlands) and Instituto de Acústica of Madrid (Spain), which is one of the institutes of the Consejo Superior de Investigaciones Científicas (CSIC). The application of the technology in washing processes was being studied by the research organisation TNO (the Netherlands), in collaboration with UT-TXT, CSIC and the Dutch industrial partners Hokatex (industrial laundry) and Van Wees (machinery producer). The University of Minho (Uminho, Portugal), the Graz University of Technology (TUGraz, Austria) and the University of Applied Sciences Cologne (UAS Cologne, Germany) were in charge of investigating the applications of ultrasound to enzymatic processes, in collaboration with the textile industry Ten Cate Advanced Textiles (Ten Cate AT, the Netherlands). The Technical University of Liberec (TUL, Czech Republic) was the main responsible of the research in the field of nonwovens. Tecnotessile (TTX) was responsible of the research carried out in the field of coating in collaboration with the Italian industrial partner Unitech (machinery producer) and Ten Cate AT; the Italian textile industry Licana has participated in this research at the beginning of the project. In the first half of the project TNO also studied the application of ultrasound to lamination, in collaboration with Multistiq, one of the Ten Cate textile companies (Ten Cate Multistiq, the Netherlands), and Forbo (adhesive producer, the Netherlands). The consortium was completed by Stimin (Italy), an ultrasound equipment manufacturer, which contributes to the development of transducers and ultrasonic equipments for novel US-based textile applications.
The consortium started executing the project activities in January 2004. According to the work carried out in the three years of the project, the research activities of the project which concern the application of ultrasound in: (1) textile washing; (2) coating with polymers and nanoparticles; (3) enzymatic processes, e.g. biodegradation of textile wastewater components; (4) nonwoven production processes; were found to be very promising in view of the achieved results and their potential for exploitation. More info about these applications is presented below per Work Package (WP) dealing with Research and Technological Development (RTD) activities.

### *1.1. WP1: Ultrasonic processing*

- *Objectives: A) Development of dedicated design rules for the intensification of textile processes using ultrasound; B) Investigation of the way in which ultrasound can affect e.g. the kinetics of enzymatic reactions, of bleach reactions and of ultrasonic coating and laminating systems; C) Development of design rules for ultrasonically driven chemical reactions in textile wet processes and ultrasonic coating and laminating operations.*

WP1 focused on gathering information knowledge and information from basic research to develop dedicated design rules for the intensification of textile processes using ultrasound, and advising partners in the activities of the other WP's where specific applications were under investigation. At the beginning of the project CSIC and UT-TXT had a meeting in Madrid during which the state of the art was identified, a strategy was developed to tackle problems, knowledge overlaps were defined and knowledge gaps in sonomechanics and sonochemistry were filled in. At the 6-month meeting in Graz UT-TXT and CSIC presented the current state of the art and their latest findings in US-based washing. UT-TXT reported the mechanisms of textile washing and the effects of the ultrasonic energy in textile washing. CSIC reported about a continuous washing system previously developed, in which textiles are exposed to the acoustic field in a flat position and in almost direct contact to the sonotrode.

In the second semester the partners started examining which are the optimal characteristics of power ultrasonic waves and the effects of non-linear phenomena associated to these waves when dealing with wet textile processes. More in details, CSIC focused on a theoretical study on the influence of non-linear phenomena on textile cleaning and mass transfer. First lab-scale tests with two different

set-ups, a US bath at 30 kHz, and a US horn at 25 kHz, were done by UT-TXT and preliminary results were obtained. Two set-ups, one working at 20 kHz, the other one at 57 kHz, were developed at CSIC to check experimentally the theoretical calculations about the production and effects of radiation pressure and acoustic streaming, under standing wave conditions, in mass transfer. Furthermore, CSIC developed a rectangular plate transducer at 20 kHz for textile cleaning, to allow the study of the production of cavitation in thin liquid layers in order to improve the continuous washing system and to develop a faster process.
Based on a literature search, an analysis was done by UT-TXT to determine how dependent and independent parameters involved in ultrasonic cleaning are interrelated and how they affect each other, with the aim to understand how these parameters can be influenced to obtain the desired effects. In the third semester CSIC studied the vibration distribution in the vibrator by the finite element method by the software ANSYS.

In the fourth semester the activities of UT-TXT concerned the influence of nucleation on washing performances. It was found that nuclei seem to have an opposite effect depending on where they were generated; nuclei inside the bulk fluid cause energy dissipation with reduction of washing performances whilst nuclei generated on the textile surface seem to promote it. CSIC constructed and tested the high power transducer designed in the previous period and also designed a semi-industrial scale ultrasonic washing machine to work in a continuous way on the basis of a former patent. Furthermore CSIC carried out one study to verify sonomechanical effects due to high intensity ultrasound waves in water. The work on this semester was focused on the characterisation of a glass washing apparatus (to minimise the wave attenuation) set-up by CSIC. A study about the acoustic fields in the reaction chamber showed that acoustic radiation force transversally distributed represents an additional force to the axial radiation force. This force brings particles distributed in the liquid in a different way in function of the contrast factor; when it is positive they are driven at the wall of a nodal plane, whilst when it is negative they are driven in the middle of an anti-nodal plane. It was observed for bubbles; they were driven to the middle of the pressure anti-node.
In the fifth semester the activities of UT-TXT were focused on two issues: (1) quantification of the results obtained by a lab-scale US-washing process at 30 kHz; (2) investigation on the Apfel's formula. A comparison between the different approaches tested so far showed that the best performance was obtained with the reflector plate. At CSIC, experiments to validate a theoretical study on the effects of two non-linear ultrasonic phenomena, radiation pressure and streaming forces, on bubbles and suspended particles in a liquid media in a standing wave field were carried out. CSIC realized a semi-industrial scale ultrasonic machine for textile washing in thin liquid layers. Interactions of undesired vibration modes were studied numerically and experimentally.
In the last semester the activities of UT-TXT focused on nucleation and sound field experiments. Hydrophone measurements were done in combination with different nuclei for the evidence of transient cavitation. The roughness of the reflector plate had no influence on the ultrasonic washing performance, which revealed that nucleation at the reflector plate did not play a role in the ultrasonic washing performance. The best ultrasonic washing performance was obtained by using a flat glass reflector plate with the smallest roughness available of 0.02 µm attached to EMPA. The inhomogeneity of the ultrasonic washing performance is caused by the ultrasonic sound field in the bath. By moving the fabric along the transducer surface and the reflector plate this inhomogeneity can be decreased. CSIC developed an ultrasonic plate transducer for textile washing that operated at 19.6 kHz. It showed a good performance at low and moderate power operation. To achieve the separation of disturbing vibrating modes from the working mode of the transducer CSIC numerically studied, constructed and experimentally tested an improved rectangular grooved-plate transducer, which vibrates at a resonant frequency of about 21.9 kHz with both faces in air and 21.1 kHz with one face submerged into water. Comparison of both transducers showed that the number of modes was dramatically reduced from six to one for the grooved-plate transducer. One important factor of the improved transducer is its stability during long-term high-power operation. The two plate transducers (19.6 and 21.1 kHz) were mounted in the ultrasonic system for textile washing

under continuous operation. The power applied varies from 400-600 W, the acoustic intensity from 3 – 5 W/cm$^2$ and the fabric speed between 0 – 1m/s. A glass reflector plate was located at the bottom of the system. The optimal distance between the reflector plate and transducer was measured by electrical conductance at 17 mm. The system allows to control and monitoring the electrical parameters of the transducers, water temperature and water level in the washing bath, the gas content of water, transducers position (distance plate-glass reflector), transducer – fabric distance, power applied and cavitation activity. Textile washing trials were done with EMPA 101 with a length of 4 m and a width of 21 cm. In the trials the fabric-plate distance was kept at about 0.5-1 mm. Fabric speed varied between 0 and 1cm/s, with a water temperature of 20ºC and the power applied to the transducers varied between 300W up to 400W. Results showed that this ultrasonic washing process was much more effective than the conventional washing machine operating at 20ºC and 50ºC (see also paragraph on WP2). The cleaning effect increased with number of transducers applied and with the power. Therefore, to increase the washing speed it will be needed to increase either the power applied or the number of transducers employed.

In addition, with the collaboration of all WP Leaders, WP1 has generated the following results as foreseen by the Description of Work: A) development of dedicated design rules for ultrasonically-driven processes in the textile industry, i.e. Deliverable D22; B) development of dedicated design rules for transducers and ultrasonic equipment for different textile processes, i.e. Deliverable D20. These two deliverables are confidential, anyhow it is to be noted that at the end of the work programme the consortium has collected all publications about the project in a public document: Deliverable D36 "Report on raising public participation and awareness", submitted to the European Commission and available on demand by contacting Tecnotessile (mail to: tecnotex@tecnotex.it).

### *1.2. WP2: Mass transfer intensification*

- *Objectives: to come to a textile rinsing process with accelerated mass transfer characteristics (by a factor of 100 to 1000) for textile cleaning.*

The partners working on WP2 were: TNO (WP Leader), UT-TXT, CSIC, Hokatex and Wan Wees.
The starting point of this research was that the introduction of pressure waves in a medium like water under certain conditions leads to cavitation: the implosion of generated gas bubbles. During this implosion, (micro)jets are generated in the direction of the centre of the gas bubble to fill the "bubble" space. In the situation that such implosion occurs on the boundary of a textile a net flow towards the textile is created (see Figure 1). The mass transfer through textiles as initiated by those microjets can be theoretically enhanced by a factor of 1000.

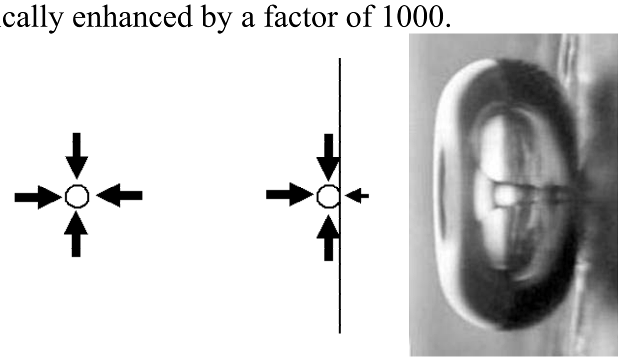


Figure 1 - When a cavitation bubble flows freely in the liquid, the micro jets that occur due to implosion are not resulting in a net flow. However near a rigid surface, e.g. textile fabric, the micro jet from the surface is negligible compared to the micro jet from the opposite direction resulting in a net flow through the fabric.

In lab-scale equipment different parameters have been evaluated which play an important role in the efficiency of the ultrasound application. Parameters investigated are, among other, time, degassed environment, nucleation, presence of chemicals and wetting of the fabric (see also the paragraph on WP1). Two continuous operating pilot machines for ultrasound-based cleaning have been constructed and different operation configurations tested. The pilot machines are shown in Figure 2 and Figure 3. The machine shown in Figure 3 is the one developed at CSIC as described in the paragraph on WP1.

Figure 2 - US-based experimental textile cleaning machine for continuous operation.

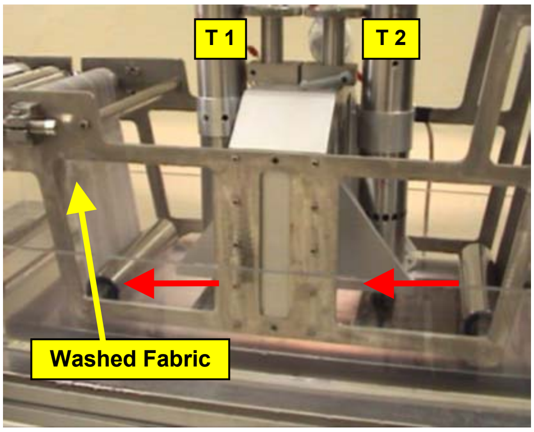



Figure 3a - Lateral-view of the core of the 2nd system. T1 and T2 represent the transducers.

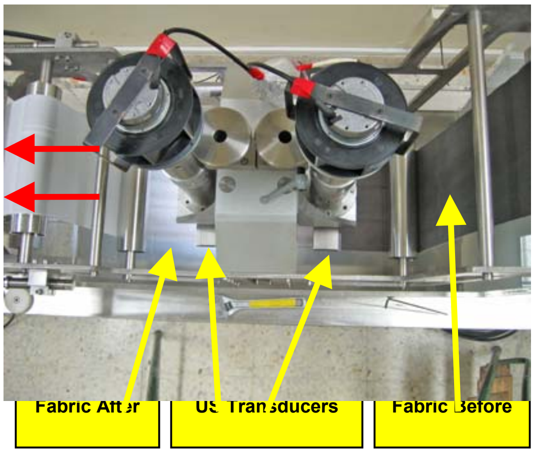


Figure 3b - Top-view of the core of the 2nd system.

As an example, a cleaning result obtained in the second pilot machine is given in Figure 4.

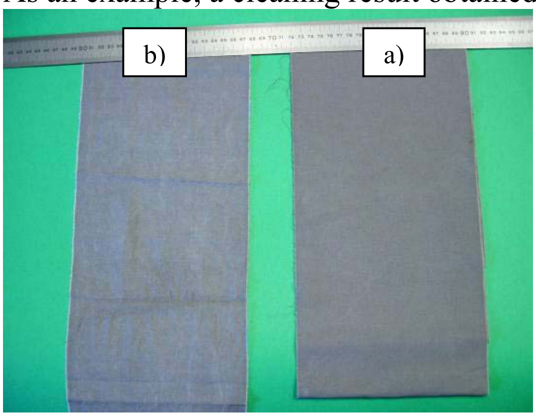



Figure 4 - a) Empa-101 reference sample. Left: b) sample after 1 conventional washing at 20ºC with commercial detergent.

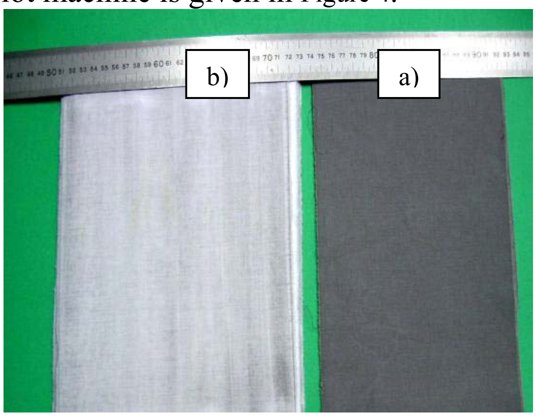



Figure 4 - a) Empa-101 reference sample. Left: b) 1 US washes 2Tx400W and commercial detergent.

Based on the experiences collected using the pilot machines, a blueprint has been prepared of a continuous operating US-based textile cleaning machine with intensified mass transfer through textiles.

### *1.3. WP3: Ultrasound coating*

- *<u>Objectives</u>: A) New processes for textiles at ambient temperature for aqueous-based and solvent-based polymer coatings; B) New processes for textiles at ambient temperature for nanoparticles-containing coatings.*

The partners working on WP3 were: TTX (WP Leader), Stimin, Unitech and Ten Cate AT.
Within the WP, sonication of various water polymer dispersions at three different frequencies (20 kHz, 60 kHz and 150 kHz) was realised by means of three transducers provided by the project partner Stimin, with the aim to determine the best condition for textile coating processes.
During lab-scale studies, the effectiveness of the tested ultrasonic processes was investigated in terms of chemical and physical changes induced by the cavitation.
It was found that the main effect in terms of coating property is an increase in the water repellence that is promoted at low frequency (20 kHz) and at low acoustic power; conversely, by increasing the applied frequency (as shown by tests at 60 and 150 kHz), a decrease in water repellence is recorded, and by increasing the power no significant changes were achieved.
The reduction of polymer particles by sonication at low frequency is responsible for this outcome, since an enhancement of the curing processes occurs thanks to an increase in low energy interactions (Van der Walls forces, hydrogen bond) induced by the increase in polar group availability.
The sonicated dispersion was then thickened with a proper chemical ad applied with a conventional coating machine on the textile surface and dried by means of a heating process.

The pre-sonication was also studied for impregnation of textiles with functional polymers such as fluorocarbon. Also in this case, a significant increase in the oil and water repellence performance was recorded. The increase of the performance for the target property is due to an increase in the availability of fluorinated groups, probably induced by the re-orientation of the polymer chains. The textile was then impregnated in the solution with a conventional padding process and the impregnated textile was dried by means of a heating process.

Sonication was also studied in order to promote the add-on of nanoparticles into different fabrics whilst the sonicator is working. Even in this case, 20 kHz was found to be the most suitable US frequency, as at this value the mass transfer reaction is promoted. An opposite behaviour in comparison with sonication of water polymer dispersions was achieved concerning the applied power: in that case, in fact, at high power a more efficient grafting of the tested inorganic particles is obtained.

Hence, on the basis of the experimental campaign on lab scale, it was concluded that 20 kHz is the best frequency for coating applications.
According to the lab tests that were realised, the horn configuration is suitable when sonication of the coating compound is performed before its application to the textile substrate, which can then be realised by means of traditional coating devices, e.g. doctor knife, but it may only be limited to small-scale processes. In fact, the horn is not suitable for sonication of high amounts of dispersions, as it is needed in case of a production on an industrial scale.

Therefore TTX, in collaboration with Unitech and Stimin, has defined two configurations to be applied for an industrial-scale use of the ultrasound technologies in coating applications and has designed and realised some pre-industrial scale machineries that have been used within the project to validate the lab-scale results. Interested parties can contact TTX for more info at: tecnotex@tecnotex.it.

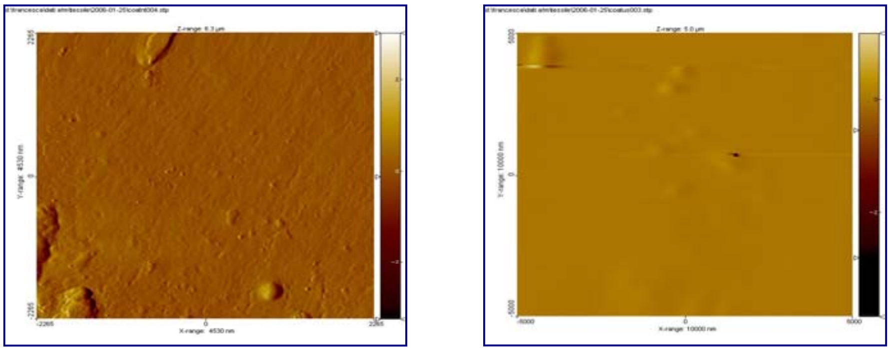


Figure 5 – Polymer coating realised with the traditional process (left) and with the application of ultrasound (right).

### *1.4. WP4: Sono-biochemistry*

- *Objectives: To study the interaction between acoustic fields and the catalytic action of enzymes in homogenous and heterogeneous systems*

The partners working on WP4 were: Uminho (WP Leader), TUGraz, UAS Cologne and Ten Cate AT.
The output of WP4 yield several public results:

**Combined ultrasound-laccase assisted bleaching of cotton.** The potential of using ultrasound to enhance the bleaching efficiency of laccase enzyme on cotton fabrics was accessed. Ultrasound of low intensity (7 W) and relatively short reaction time (30 min) seems to act in a synergistic way with the enzyme in the oxidation/removal of the natural colouring matter of cotton. The increased bleaching effect could be attributed to improved diffusion of the enzyme from the liquid phase to the fibres surface and throughout the textile structure. On the other hand inactivation of the laccase occurred increasing the intensity of the ultrasound. However, at the ultrasound power applied in the bleaching experiments the loss of enzyme activity was not significant enough to justify the use stabilizer such as polyvinyl alcohol. Furthermore, the polyvinyl alcohol appears to be a substrate for the laccase[1].

**Stability and decolourization ability of *Trametes villosa* laccase in liquid ultrasonic fields.** The sonication of laccase from *Trametes villosa* and bovine serum albumin promotes the formation of protein aggregates with high molecular weight. The formation of aggregates leads to the deactivation of the enzyme, fact that was confirmed by the analysis of the enzyme stability (half-life time) upon ultrasound treatment. This inactivation was mainly caused by the radicals formed by the cavitation phenomenon. It was verified that the addition of polyvinyl alcohol to laccase had a protecting effect against enzyme inactivation. The performance of laccase in the decolourization of indigo carmine was studied. It was observed that the best results were attained when the dye solution was treated with ultrasound and enzyme stabilized with polyvinyl alcohol, where more than 65% of decolourization was achieved. This value is remarkably higher than that attained for the

[1] *Carlos Basto et al. Ultrasonics Sonochemistry, Volume 14, Issue 3, March 2007, Pages 350-354.*

enzyme alone, which was only able to decolourize 20% of the dye solution within 1 h of treatment. These results have important implications for the exploitation of sonication in textile industry, where the pollution caused by the release of dyes into effluents is one of the major concerns[2].

**Staining of wool using the reaction products of ABTS oxidation by Laccase: Synergetic effects of ultrasound and cyclic voltammetry.** The effects of ultrasound on 2,2′-Azinobis(3-ethylbenzothiazoline-6-sulfonate) enzymatic oxidation by laccase (*Trametes villosa*) has been studied by means of cyclic voltammetry. The reaction was allowed to proceed in the presence of a piece of wool and the coloration depth of the wool fabric was measured by means of *K*/*S*. It was observed that cyclic voltammetry is influenced the dyeing process and higher *K*/*S* values were obtained when the cyclic voltammetry was combined with the ultrasonic irradiation. Moreover, the *K*/*S* value is the sum of the values obtained when the wool staining is done in just the presence of cyclic voltammetry or in just the presence of ultrasound. The results obtained on the indigo carmine decolourization gives information on the importance of controlling the amount of $ABTS^{+}$ formed during the ultrasonication process[3].

**Application of power ultrasound for azo dye degradation.** Power ultrasound of 850 kHz at 60, 90 and 120 W was used for the degradation of industrial azo dyes Acid Orange 5 and 52, Direct Blue 71, Reactive Black 5 and Reactive Orange 16 and 107. The results show that power ultrasound is able to mineralize azo dyes to non-toxic end products, which was confirmed by respiratory inhibition test of *Pseudomonas putida*. All investigated dyes have been decolorized and degraded within 3–15 h at 90 W and within 1–4 h at 120 W, respectively. Mass spectrometric investigations show, that hydroxyl radicals attack azo dyes by simultaneous azo bond scission, oxidation of nitrogen atoms and hydroxylation of aromatic ring structures. A volumetric scale-up showed a correlation between the energy input and the absolute amount of degraded dye. Up to an energy input of about 90 W no enzymatic deactivation of laccase was observed which might be helpful for a simultaneous action of sonochemical and enzymatic treatments[4].

**Degradation of Azo Dyes by Laccase and Ultrasound Treatment.** The goal of this work was to investigate the decomposition of azo dyes by oxidative methods, such as laccase and ultrasound treatments. Each of these methods has strong and feeble sides. The laccase treatment showed high decolorization rates but cannot degrade all investigated dyes (reactive dyes), and high anionic strength led to enzyme deactivation. Ultrasound treatment can decolorize all tested dyes after 3 h at a high energy input, and prolonged sonication leads to nontoxic ionic species, which was demonstrated by ion chromatography and toxicity assays. For the first time, it was shown that a combination of laccase and ultrasound treatments can have synergistic effects, which was shown by higher degradation rates. Bulk light absorption and ion-pairing high-performance liquid chromatography (IP-HPLC) were used for process monitoring, while with reversed-phase HPLC, a lower number of intermediates than expected by IP-HPLC was found. Liquid chromatography-mass spectrometry indicated that both acid orange dyes lead to a common end product due to laccase treatment. Acid Orange 52 is demethylated by laccase and ultrasound treatment. Further results confirmed that the main effect of ultrasound is based on $^{\cdot}OH$ attack on the dye molecules[5].

### *1.5. WP5: Ultrasound application for nonwovens*

*__Objectives__: A) Obtaining US-crosslinked super-absorbent polymers to create more effective absorption and hence to provide users with more comfort; B) To improve the binder uptake and*

[2] Carlos Basto et al.Ultrasonics Sonochemistry, Volume 14, Issue 3, March 2007, Pages 355-362.
[3] Florentina-Daniela Munteanu et al. Ultrasonics Sonochemistry, Volume 14, Issue 3, March 2007, Pages 363-367.
[4] Astrid Rehorek et al. Ultrasonics Sonochemistry, Volume 11, Issues 3-4, May 2004, Pages 177-182.
[5] Michael M. Tauber et al. Applied and Environmental Microbiology, May 2005, p. 2600-2607, Vol. 71, No. 5.

*to make easier dislocation of polymer particles during and after wet nonwovens bonding, to obtain textiles with improved mechanical properties.*

The partner that had the leadership of this WP was TUL. The Czech partner has widely investigated: A) sonocatalysis and sonochemistry for Super Absorbent Polymer (SAP) fibres; B) application of ultrasound to chemical bonding of nonwoven webs.

**Sonocatalysis and sonochemistry for SAP fibres.**
Ultrasound was used for ultrasonic irradiation of crosslinking reaction in SAP nanofibers web, where the crosslinking initiator was included.
A solution of different crosslinking agents was dosed from an infuse set on sonotrode surface and sprayed into fine aerosol. The aerosol uniformly moisturized completely on the surface of nanofiber web and it enabled the incidence of crosslinking agents without dipping of crosslinking system. The ultrasonic treatment with frequency 30 kHz and power 60 W was employed for this experiment. In some cases it was necessary to use consequent heat treatment.
A special atomizing set-up has been used for application of crosslinking agent. The ultrasonic atomizer (nebulizer) is a device that turns liquids into fine spray. The professional atom disintegrator prepares aerosol with very fine drops, the mist. It uses mechanical forces from an oscillating transducer to break a liquid into very small droplets with a relatively uniform droplet size. The system allows precise control of the energy at the nebulizing transducer and provides an extensive range of ultrafine spray patterns to enhance liquid dispersion, penetration, and wetting characteristics. A closely regulated volume of liquid with catalytic agent can be atomized and deposited on virtually any porous substrate as a uniform reproducible humectation. The mist produced in this way was sucked through a porous material by a ventilator. This set-up enables active application of catalytic agent on through the whole material. The advantage of this application of catalytic agents was low wetting of substrate and consequent low energy consumption for drying. The special ultrasonic set-up for application of crosslinking agent on the nanofiber web was constructed. Influence of temperature and surface tension of solution was tested. Different crossslinking agents or initiating agent from water solution were tested for nanofiber webs. Nanofibers from pHEMA (copolymer 2-hydroxyethyl methacrylate and natrium salt methyl acrylate, from a blend of polyvinylalcohol and chitosan were treated by ultrasonic generated mist. Using SEM images the effect of crosslinking and subsequent fiber stability against water solution after water treatment were controlled. Positive influence of non-water solutions for crosslinking agents was expected. There are many parameters, which influenced quantity and quality (droplet formation) nebulized mist: (i) type of liquid, (ii) input power, (iii) the height of liquid level above atomizer, (iiii) composition of mixture, etc. The dependence of nebulizing process on the composition of mixture was studied. Great progress in comprehension of organic liquid atomizing behavior was achieved.

Glycerol was chosen as a crosslinking agent for polyacrylic acid nanofibers. Properties of this trihydric alcohol do not allow a nebulization process. Suitable mixture of organic liquids (glycerol-methanol – acetone), which is enough resistant to polyacrylic nanofiber sheet, was found.

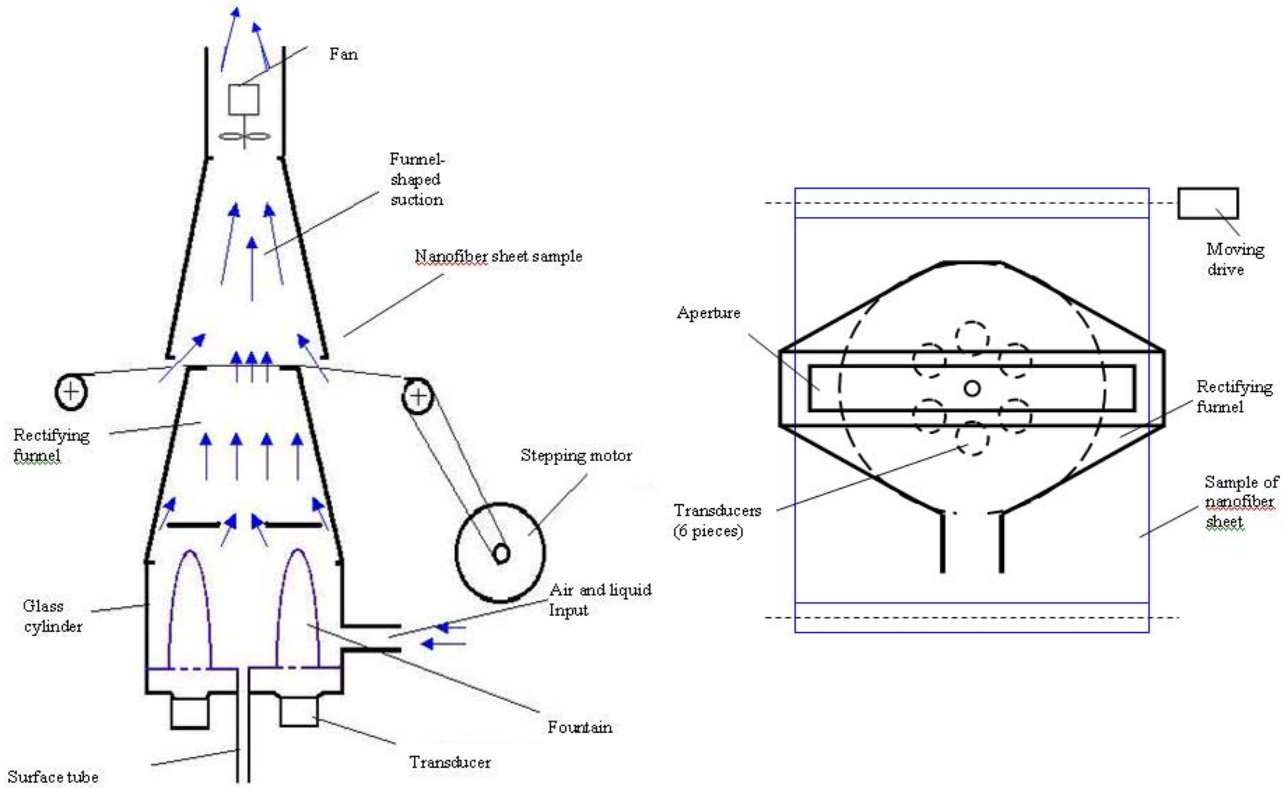



Figure 6 - Section plan of continual nebulizer „Mlhos3".

Construction of pilot plant continual equipment for nebulization of nanofiber sheets with organic liquid was solved. This functional model works with one to maximal six transducers, so intensity of processed mist could be fully controllable.
A very effective continual nebulizer „Mlhos 3" was designed (see Figure 6). This equipment was furnished with six piezoelectric transducers that increased the effectiveness minimally six times. The body of nebulizer was constructed from chemical inactive glass, which allows working with all organics liquids.

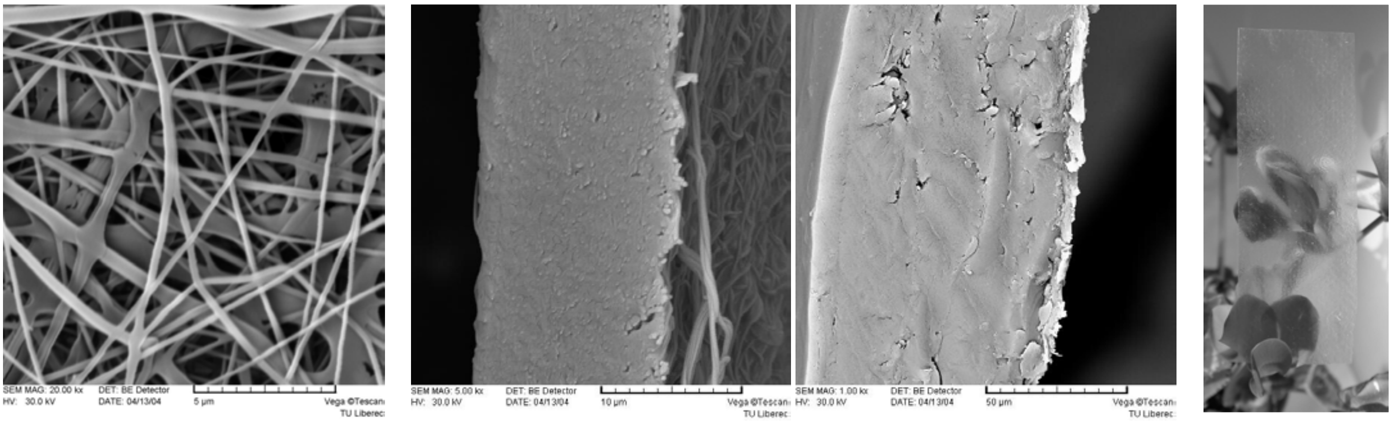
The following publications about this research were finalised during the project:

- L. Martinova, P. Hana (2004). Stabilization of nanofibers using ultrasonic nebulizer. Proceedings of the $11^{th}$ International Conference STRUTEX, Liberec, Czech Republic, 6-7 December.
- L. Martinová, P. Pokorný (2006). Crosslinking of nanofibers using ultrasonic generated mist. Book of abstracts of the $10^{th}$ Meeting of the European Society of Sonochemistry, Hamburg, Germany, 4-8 June.

**Application of ultrasound to chemical bonding of nonwoven webs.**
The task was split to several branches according to concerns of co-workers from TUL. Here we report the public results of some of these research studies.

First the research was focused on a binding of nanofiber webs by latex and resin matrixes by means of ultrasound (see Figure 7) or we can say on a manufacturing of composite materials reinforced by electrospun nanofibers.

Figure 7 – From left: SEM picture of a basic nanofiber web (a), a cross-sections of the fully filled (b) and partially filled- strengthened (c) nanofibre webs by latex's matrix and photo of final transparent composite material (d).



According to the results that were obtained, the technology of impregnation of nanofiber layers for production of composite materials using ultrasound seems to be very promising. The main success is the high content of fibers in composites. This research yielded the following publications:

- E. Kostakova (2004). Nanofibers as reinforcement in composites. Proceedings of Reinforced Plastics 2004, Balatonvilágos, Hungary, 25-27 May.
- L. Ocheretna, E. Kostakova (2005). Ultrasound and textile technology – cellular automata simulation and experiments. Proceedings of Forum Acusticum conference, Budapest, Hungary, 29 August – 2 September.

Some interesting results were also obtained with a research study on electrospinning technology enhanced by ultrasound. The free liquid surface of polyvinyl alcohol (PVA) polymer solutions was submitted simultaneously to an electrical field and ultrasound vibrations. In electrospinning the omnipresent perturbations on the liquid surface, which are called capillary waves, provoke a fluctuation on the liquid surface that is magnified by the external electrostatic field. These conical objects are called Taylor cones from which either fibrous jets or droplets are pulled. Capillary waves on a liquid surface can be influenced by high-frequency ultrasonic waves. Correlation between both phenomena was demonstrated within the project (S. Torres, D. Lukas (2005). Ultrasound enhanced needle-less Electrospinning. Proceedings of the conference NANO'05, Brno, Czech Republic, 8 – 10 November).

Another major research topic of TUL concerned the penetration of powder and granular materials into nonwovens mediated by ultrasound. Problems with penetration of these materials in a textile or nonwoven substrate is connected with the high density and thickness of the substrate. Pores in a textile or a nonwoven are too little for penetration of powder or granular materials. TUL found that the ultrasound treatment can improve the powder penetration process (Novák Ondřej (2005). Powder penetration of bulky nonwovens by the help of ultrasound. Proceedings of the 5th AUTEX conference, Portoroz, Slovenia, 27-29 June). Accordingly, ultrasound was successfully used for the penetration of granular materials in the structure of nonwovens with the aim of developing bulletproof materials. A publication concerning this work was finalized:

- O. Novak, D. Lukas (2006). Bullet-proof vests from granular materials? Proceedings of Autex 2006 Conference, Raleigh, USA, 11-14 June.

A special device for the US-mediated application of powder or granular materials to nonwovens was constructed by TUL. Scheme is visible in the figure below.

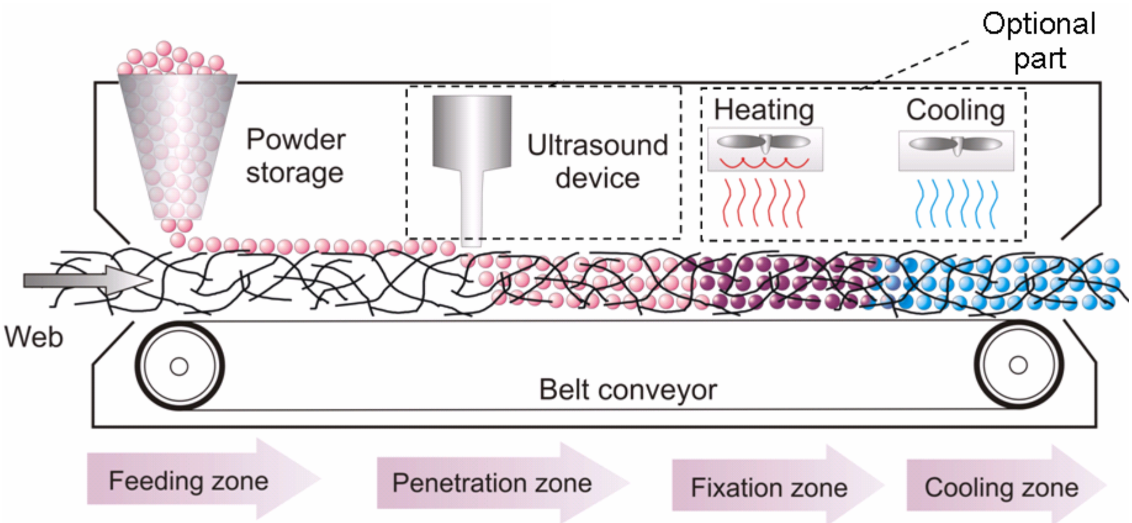



Figure 8 - Scheme of device for ultrasound-mediated application of powders or granular materials to nonwovens.

## 2. Dissemination and use

According to the EC document "*Project reporting in FP6. Guidance notes for Integrated Projects, Networks of Excellence, Specific Targeted Research or Innovation Projects, Coordination Actions, Specific Support Actions, Co-operative Research Projects and Collective Research Projects*" issued on October 2004, page 15, this chapter is equal to the section "Publishable results" of the Final Plan for Using and Disseminating the Knowledge. Hence, we refer to the latter for the related info.