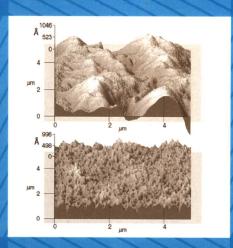
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Plasma technologies for textiles

Edited by R. Shishoo







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Contributor contact details

(* = main contact)

Editor

Professor Roshan Shishoo Director Shishoo Consulting AB Svartlavsvägen 18 SE-436 39 ASKIM Sweden

E-mail: roshan.shishoo@ shishooconsulting.se

Chapter 1

Bill Graham
Professor of Physics
Director of IRCEP (The
International Research Centre for
Experimental Physics)
Department of Physics and
Astronomy
Queen's University Belfast
BT7 1 NN
Northern Ireland, UK

E-mail: b.graham@qub.ac.uk

Chapter 2

Professor James W. Bradley* and Dr Paul M. Bryant Department of Electrical Engineering and Electronics University of Liverpool Brownlow Hill Liverpool L69 3GJ UK

E-mail: j.w.bradley@liverpool.ac.uk

Chapter 3

Paul Lippens
Sales and Marketing Manager
Europlasma N.V.
De Bruwaan 5/d
B-9700 Oudenaarde
Belgium

E-mail: paul.lippens@europlasma. be

Chapter 4

Tony Herbert
Marketing Manager
Dow Corning Plasma Solutions
Unit 12 Owenacurra Business Park
Midleton
Co. Cork
Ireland

E-mail: tony.herbert@dowcorning.

Chapter 5

Dr Thomas Stegmaier*, Dr Alexander Rau, Albrecht Dinkelmann and Dr Volkmar von Arnim Institute for Textile Technology and Process Engineering Denkendorf Denkendorf Center of Excellence for Technical Textiles Koerschtalstrasse 26 D-73770 Denkendorf Germany

E-mail: thomas.stegmaier@ itv-denkendorf.de

Chapter 6

Dr Dirk Hegemann* and Dr Dawn Balazs Empa – Materials Science and Technology, Advanced Fibers Lerchenfeldstrasse 5 9014 St. Gallen Switzerland

E-mail: dirk.hegemann@empa.ch

Chapter 7

Dr Stephen Coulson Technical Director P2i Ltd Unit 14, Central 127 Milton Park, Abingdon Oxfordshire OX14 4SA UK

E-mail: Stephen.coulson@p2ilabs.com

Chapter 8

Dr Uwe Vohrer Fraunhofer Institute for Interfacial Engineering and Biotechnology Nobelstrasse 12 70569 Stuttgart Germany

E-mail: uwe.vohrer@igb.fraunhofer. de

Chapter 9

Dr Helga Thomas DWI at the RWTH Aachen e.V. Pauwelsstrasse 8 D-52056 Aachen Germany

E-mail: Thomas@dwi.rwth-aachen. de

Chapter 10

Kenth Johansson
Area Manager Surface
Modification
Materials and Coatings section
YKI Ytkemiska Institutet AB
Box 5067
SE -114 86 Stockholm
Sweden

E-mail: kenth.johansson@surfchem. kth.se

Chapter 11

Bruno Marcandalli* and Claudia Riccardi Stazione Sperimentale per la Seta Via Giuseppe Colombo 83 20133 Milano Italy

E-mail: marcandalli@ssiseta.it

Chapter 12

Professor Anne Neville*
Professor of Tribology and Surface
Engineering
School of Mechanical Engineering
University of Leeds
Leeds
LS2 9JT
UK

E-mail: a.neville@leeds.ac.uk

Dr R.R. Mather and Professor J.I.B. Wilson School of Textiles and Design Heriot-Watt University Edinburgh EH14 4AS UK

Introduction – The potential of plasma technology in the textile industry

R. SHISHOO Shishoo Consulting AB, Sweden

Since their introduction in the 1960s, the main industrial applications of (low-pressure) plasmas have been in the micro-electronic industries. In the 1980s their uses broadened to include many other surface treatments, especially in the fields of metals and polymers. The dominant role of plasmatreated surfaces in key industrial sectors, such as microelectronics, is well known, and plasmas, certainly experimentally and, in places, industrially, are being used to modify a huge range of material surfaces, including plastics, polymers and resins, paper and board, metals, ceramics and inorganics, and biomaterials. Properties enhanced include wettability, adhesion, biocompability, protection and anti-wear, sterilisation, and chemical affinity or inertness. The prospects of very good technical and economical results, as experienced in the microelectronics industry, are stimulating efforts worldwide to apply plasma processing more widely to the processing of textiles and nonwovens. Undoubtedly, tremendous advantages are afforded by plasma technology as a uniquely effective engineering tool for achieving, in a flexible and versatile way, a broad range of functionalisations of textiles and nonwovens.

In the textile field, significant research work has been going on since the early 1980s in many laboratories across the world dealing with low-pressure plasma treatments of a variety of fibrous materials showing very promising results regarding the improvements in various functional properties in plasma-treated textiles. A variety of commercial low-pressure plasma machines, mostly in prototype form, have been offered for batch/in-line processing of textiles for more than 15 years. In recent times, some companies have also started to offer commercial systems for atmospheric-pressure plasma processing of textiles, both in-line and on-line. Despite all the significant benefits demonstrated in the laboratory and industrial prototypes, plasma processing on an industrial scale has been slow to make an impact in the textile industry. This may be due to factors such as important gaps in the relevant applied research, slow development of suitable industrial plasma systems, late focus on developing in-line atmospheric pressure

plasma systems and less public transparency regarding the successes and failures of industrial trials.

The textile and clothing industries in Europe, North America and some other developed countries are facing some big challenges today, largely because of the globalisation process. Therefore, the shift to high-functional, added value and technical textiles is deemed to be essential for their sustainable growth. The growing environmental and energy-saving concerns will also lead to the gradual replacement of many traditional wet chemistry-based textile processing, using large amounts of water, energy and effluents, by various forms of low-liquor and dry-finishing processes. Plasma technology, when developed at a commercially viable level, has strong potential to offer in an attractive way achievement of new functionalities in textiles. In recent years, considerable efforts have been made by many plasma technology suppliers to develop both low-pressure and atmospheric-pressure based plasma machinery and processes designed for industrial treatment of textiles and nonwovens to impart a broad range of functionalities.

What are plasmas?

The coupling of electromagnetic power into a process gas volume generates the plasma medium comprising a dynamic mix of ions, electrons, neutrons, photons, free radicals, meta-stable excited species and molecular and polymeric fragments, the system overall being at room temperature. This allows the surface functionalisation of fibres and textiles without affecting their bulk properties. These species move under electromagnetic fields, diffusion gradients, etc. on the textile substrates placed in or passed through the plasma. This enables a variety of generic surface processes including surface activation by bond breaking to create reactive sites, grafting of chemical moieties and functional groups, material volatilisation and removal (etching), dissociation of surface contaminants/layers (cleaning/scouring) and deposition of conformal coatings. In all these processes a highly surface specific region of the material (<1000 Å) is given new, desirable properties without negatively affecting the bulk properties of the constituent fibres.

Plasmas are acknowledged to be uniquely effective surface engineering tools due to:

- Their unparalleled physical, chemical and thermal range, allowing the tailoring of surface properties to extraordinary precision.
- Their low temperature, thus avoiding sample destruction.
- Their non-equilibrium nature, offering new material and new research areas.
- Their dry, environmentally friendly nature.

Plasma reactors

Different types of power supply to generate the plasma are:

Low-frequency (LF, 50–450 kHz)

Radio-frequency (RF, 13.56 or 27.12 MHz) Microwave (MW, 915 MHz or 2.45 GHz)

The power required ranges from 10 to 5000 watts, depending on the size of the reactor and the desired treatment.

Low-pressure plasmas

Low-pressure plasmas are a highly mature technology developed for the microelectronics industry. However, the requirements of microelectronics fabrication are not, in detail, compatible with textile processing, and many companies have developed technology of low pressure reactors to achieve an effective and economically viable batch functionalisation of fibrous products and flexible web materials.

A vacuum vessel is pumped down to a pressure in the range of 10^{-2} to 10^{-3} mbar with the use of high vacuum pumps. The gas which is then introduced in the vessel is ionised with the help of a high frequency generator. The advantage of the low-pressure plasma method is that it is a well-controlled and reproducible technique.

Atmospheric pressure plasmas

The most common forms of atmospheric pressure plasmas are described below.

Corona treatment

Corona discharge is characterised by bright filaments extending from a sharp, high-voltage electrode towards the substrate. Corona treatment is the longest established and most widely used plasma process; it has the advantage of operating at atmospheric pressure, the reagent gas usually being the ambient air. Corona systems do have, in principle, the manufacturing requirements of the textile industry (width, speed), but the type of plasma produced cannot achieve the desired spectrum of surface functionalisations in textiles and nonwovens. In particular, corona systems have an effect only in loose fibres and cannot penetrate deeply into yarn or woven fabric so that their effects on textiles are limited and short-lived. Essentially, the corona plasma type is too weak. Corona systems also rely upon very small interelectrode spacing (~1 mm) and accurate web positioning, which are incompatible with 'thick' materials and rapid, uniform treatment.

Dielectric barrier discharge (Silent discharge)

The dielectric barrier discharge is a broad class of plasma source that has an insulating (dielectric) cover over one or both of the electrodes and operates with high voltage power ranging from low frequency AC to 100 kHz. This results in a non-thermal plasma and a multitude of random, numerous arcs form between the electrodes. However, these microdischarges are non-uniform and have potential to cause uneven treatment.

Glow discharge

Glow discharge is characterised as a uniform, homogeneous and stable discharge usually generated in helium or argon (and some in nitrogen). This is done, for example, by applying radio frequency voltage across two parallel-plate electrodes. Atmospheric Pressure Glow Discharge (APGD) offers an alternative homogeneous cold-plasma source, which has many of the benefits of the vacuum, cold-plasma method, while operating at atmospheric pressure.

Summary

Cold plasmas can be used for various treatments such as: plasma polymerisation (gaseous monomers); grafting; deposition of polymers, chemicals and metal particles by suitable selection of gas and process parameters; plasma liquid deposition in vaporised form.

Gases commonly used for plasma treatments are:

- Chemically inert (e.g. helium and argon).
- Reactive and non-polymerisable (e.g. ammonia, air, and nitrogen).
- Reactive and polymerisable (e.g. tetrafluoroethylene, hexamethyldisiloxane).

Effect of plasma on fibres and polymers

Textile materials subjected to plasma treatments undergo major chemical and physical transformations including (i) chemical changes in surface layers, (ii) changes in surface layer structure, and (iii) changes in physical properties of surface layers. Plasmas create a high density of free radicals by disassociating molecules through electron collisions and photochemical processes. This causes disruption of the chemical bonds in the fibre polymer surface which results in formation of new chemical species. Both the surface chemistry and surface topography are affected and the specific surface area of fibres is significantly increased. Plasma treatment on fibre and polymer surfaces results in the formation of new functional groups such as —OH, —C=O, —COOH which affect fabric wettability as well as facilitate graft

polymerisation which, in turn, affect liquid repellence of treated textiles and nonwovens.

Adhesion problems, especially for synthetic fibre-based fabrics, often arise in coating, bonding and printing of textile materials. The characteristic low surface energy of many polymeric substrates results in intrinsically poor adhesion. Adhesion is fundamentally a surface property, often governed by a layer of molecular dimensions. Many types of wet-chemical surface treatments for adhesion enhancement are becoming increasingly unacceptable because of environmental and safety considerations. Modification of polymer and fibre surfaces by plasma treatment presents many important advantages and offers a great potential as an established means of fabric processing.

In the plasma treatment of fibres and polymers, energetic particles and photons generated in the plasma interact strongly with the substrate surface, usually via free-radical chemistry. Four major effects on surfaces are normally observed. Each is always present to some degree, but one may be favoured over the others, depending on the substrate and the gas chemistry, the reactor design, and the operating parameters. The four major effects are surface cleaning, ablation or etching, cross-linking of near-surface molecules and modification of surface-chemical structure. All these processes, alone or in synergistic combination, affect adhesion. Plasma treatment can be used with great effect to improve the bond strength of polymer to fibre and polymer to polymer combinations. In these cases, the improved adhesion results from both increased wettability of the treated substrate and the modification of surface chemistry of the polymer.

Modified wettability is one of the most apparent results of plasma treatment and the method used for characterising the modification is to measure the advancing and receding contact angles against specific liquids. Plasma-produced polar groups increase the surface free energy, γ , of the fibre and decrease the contact angle, θ , usually correlating with better bonding of adhesives; θ has often been used as an estimate of bonding quality.

Plasmas offer uniquely effective surface engineering tools due to their unparalleled physical, chemical and thermal range, their low temperature (avoiding sample destruction), their dry, environmentally friendly nature, and their nonequilibrium nature offering new material and new research areas.

The advantages of an industrially viable plasma processing system over traditional textile processing are summarised in Table 0.1.

Plasma finishing of textiles

An excellent state-of-the art description of plasma technology and plasma treatment for textiles was presented in six papers in the session on plasma finishing at the TECHTEXTIL – Symposium, May 1997.

Table 0.1 Plasma treatment vs. traditional textile processing

	Plasma processing	Traditional wet chemistry
Medium	No wet chemistry involved. Treatment by excited gas phase	Water-based
Energy	Electricity – only free electrons heated (<1% of system mass)	Heat – entire system mass temperature raised
Reaction type	Complex and multifunctional; many simultaneous processes	Simpler, well established
Reaction locality	Highly surface specific, no effect on bulk properties	Bulk of the material generally affected
Potential for new processes	Great potential, field in state of rapid development	Very low; technology static
Equipment	Experimental, laboratory and industrial prototypes; rapid industrial developments	Mature, slow evolution
Energy consumption	Low	High
Water consumption	Negligible	High

Within the scope of an EU-project LEAPFROG CA, an extensive literature analysis and patent survey was carried out in 2005 in the area of plasmas and plasma-induced functionality of textiles. Hundreds of articles have been written on these subjects and a very large number of patents have been granted in the field of plasma treatment of fibres, polymers fabrics, nonwovens, coated fabrics, filter media, composites, etc. for enhancing their functions and performance. This survey has pointed out the potential use of plasma treatments of fibres, yarns and fabrics for the following types of functionalisation:

- Anti-felting/shrink-resistance of woollen fabrics.
- Hydrophilic enhancement for improving wetting and dyeing.
- Hydrophilic enhancement for improving adhesive bonding.
- Hydrophobic enhancement of water and oil-repellent textiles.
- Facilitating the removal of sizing agents.
- Removing the surface hairiness in yarn.
- Scouring of cotton, viscose, polyester and nylon fabrics.
- Anti-bacterial fabrics by deposition of silver particles in the presence of
 -plasma.
- Room-temperature sterilisation of medical textiles.

- Improved adhesion between textiles and rubber.
- Plasma-treated fabrics with high hydrophilic stability when stored in alkaline media.
- Graft plasma polymerisation for producing fabrics with laundry-durable oleophobic, hydrophobic and stain-resistant finishes.
- Atmospheric plasma-based graft polymerisation of textiles and nonwovens having different surface functional properties on the face and back side of the fabric.
- A fabric which is coated with sizing agent inactive to plasma on one side and on the other side left as hydrophobic or hydrophilic after size removal, the resultant fabric having different functionality on its two sides.
- Flame-retardant coating using monomer vapour (halogen and/or phosphorus) in combination with nitrogen and/or silicone.
- Silicone coating of air-bag fabrics using crosslinked silicone (polyorganosiloxanes).
- Scouring of cotton, rayon, polyester fabrics using a non-polymerisable gas (nitrogen, argon, ammonia, helium), followed by wet treatment for removing the impurities.
- Prevention of readily-occurring colour variation in textiles.
- Durable antistatic properties using PU-resin and plasma processing.
- Shrink resistance of animal hair textiles using urethane-based resin and plasma processing.
- Electro-conductivity of textile yarns by surface plasma deposition.

Plasma systems for experimental and industrial processing of fabrics

Until recently the only available plasma systems for textiles were low-pressure systems designed for batch operation. This type of plasma machinery also exists for 'air-to-air' or 'cassette-to-cassette', batch—continuous in-line systems for yarn, film and fabric treatments. However, low-pressure plasma treatment is essentially a batch process with fabric being treated as it is wound from one batch to another inside a vacuum chamber.

There is a great need for commercially viable plasma systems able to perform continuous on-line treatment of fabrics and films for finishing and coating applications. For this purpose both the low-pressure and the atmospheric-pressure plasma systems would be suitable for processing of fibres, yarns and fabrics. Whereas some commercial low-pressure plasma systems have been around for a few years, lately there has been a push by some plasma machinery manufacturers to build atmospheric-pressure based plasma systems for on-line and in-line use and to market these for different types of functionalisation.

The industrial objectives for these plasma systems have been to produce plasma reactors and fabric-handling systems able to process continuous, roll-to-roll lengths of fabrics, process widths up to 5 metres or more, process at speeds of greater than 10 metres per minute, process at or near room temperature and obtain uniform, deep-penetration and surface modification of fibres in textiles and nonwovens, and be capable of handling large fabrics with wide electrode spacing.

Despite the proven capability and flexibility of plasma technology in the laboratory environment and despite very significant market potentials, the commercial use of the available plasma systems for industrial processing of textiles is still almost none. The systems offered in the European market are:

Low-pressure plasma systems capable of:

In-line treatment and batch treatment: Europlasma (Belgium), P2i (UK), Mascioni (Italy)

Atmospheric-pressure plasma systems capable of:

On-line treatment and batch treatment. On-line treatment and continuous systems: Dow Corning Plasma Systems (Ireland), Ahlbrandt (Germany), AcXys (France)

These companies offer standard and custom-designed machine construction as well as specially designed laboratory plasma units. Some companies have provided proprietary liquid precursor delivery/deposition systems. P2i has developed plasma chambers for treating end-products such as clothing.

Cost considerations

The cost of energy, chemicals and cooling water used in plasma treatments is relatively low. However, there is a big investment in machinery and personnel. The crucial factors for the customer company are expected utilisation rates and projected cycle times.

Plasma technology for textile finishing: Barriers and challenges

Although it has long been verified in laboratory and industrial plasma prototypes around the world that plasma technology is a versatile and environmentally friendly finishing process for achieving a broad spectrum of textile functionalisation, its application due to some known and unknown reasons has still not gathered commercial momentum. Some of these reasons may include important gaps in the relevant applied research, slow develop-

ment of suitable industrial plasma systems, a late focus on developing on-line atmospheric pressure plasma systems and less public transparency regarding the successes and failures of industrial trials.

It is well known that all plasma machinery manufacturers do carry out interesting product development work for many textile and nonwoven companies. However, this work is mainly carried out under strict confidentiality agreements and consequently the successes and failures of these activities are not known in public. It is the editor's strong belief that the commercial application of plasma technology in the textile industry will gather momentum and will become widespread if there is more transparency as regards results and experience of textile companies that are successfully using this technology in their production.

About the book

The objective of this book is to give to the reader a comprehensive description of the science and technology related to plasmas, with particular emphasis on their potential use in the textile industry. The book contains, probably for the first time ever, a collection of the essential knowledge and information about plasmas as a smart engineering tool for obtaining the desired surface characteristics on fibres essential for producing high-functional and added value textiles, technical textiles and nonwoven products. The contributors to this book represent a team of international experts at the cutting edge of plasma technology and high-functional textiles.

In Chapter 1, Bill Graham gives a detailed description of the physics and chemistry of plasmas and describes how the unique physical and chemical characteristics of the plasma environment make it attractive for textile processing. The physical characteristics of plasmas are described in this chapter, along with the general chemical characteristics and surface interactions of partially ionised gases. The basic properties of the gases in which plasmas are formed, as well as the basic concepts of the creation of plasmas and their physical structure, are discussed along with the conditions that must prevail for an ionised gas to behave as plasma and the parameters that describe the plasma. The author then describes in detail the unique aspects of plasma chemistry with regard to the constituent species, their collisions and interactions. In textile processing, the interactions of the plasma generated species with and on the surfaces in contact with the plasma is of great importance and these are also discussed in this chapter.

In Chapter 2, James Bradley and Paul Bryant describe the diagnosis and control of plasma parameters in the processing of textiles and other materials. Plasma diagnostic tools are a useful, if not essential, element towards the proper understanding and development of technological plasmas. Knowledge of the particle densities and energies in the bulk and

at boundaries, the electrical potentials in the system and the spatial and temporal evolution of all of these parameters allow physicists and technologists not only to operate plasmas in the most efficient way but also to allow the plasma intrinsic processes to be tailored to suit a particular process. The authors then describe various types of diagnostic tools that can be used depending on the type of plasma under investigation and the specific information that is required. The following four techniques are described in detail: (i) discharge electrical characteristics, i.e. the nature of the driving currents and voltages of a plasma discharge and their relationship, which can provide much information about the bulk plasma properties (interpretation of the driving current and voltage waveforms yields information on the major processes in the discharge); (ii) electric plasma diagnostics based on the electrical properties of plasmas, which are perhaps the most frequently used methods of determining the local plasma parameters such as electron density, N_{eo} , temperature, T_e , and plasma potential, V_p ; (iii) plasma mass spectrometry, which is a well-developed technique that provides information on the identity of the neutral and charged species present in a discharge, and their relative fluxes to a material surface or substrate. The technique is also an ideal tool to understand the behaviour of plasmas with complicated mixtures of chemical precursors; (iv) optical emission spectroscopy. This section attempts to cover of the important aspects of optical emission spectroscopy and its use as a diagnostic. An exhaustive list of references has been included in this chapter.

In Chapter 3, Paul Lippens describes the low-pressure cold plasma technology for treatment of textiles and nonwovens. Description is given of the low-pressure vacuum plasma systems for plasma activation, plasma etching and plasma polymerisation of fabrics. The author then describes some existing equipment for low-pressure vacuum plasma technology including web treatment equipment based on box-type vacuum chambers as well as some roll-to-roll web treatment equipment. This is followed by presentation of results showing the benefits of vacuum plasma treatment on fabrics for automotive and medical applications, plasma pre-treatment prior to dyeing, activation of automotive textiles before application of flame retardant chemistry and hydrophobic nonwovens for filtration applications. The author mentions that, for most of these applications, industrial size low-pressure plasma machinery has already been manufactured and used. The author also discusses the economics of vacuum plasma treatment for fabrics and nonwovens.

In Chapter 4, Tony Herbert gives a comprehensive description of various types of atmospheric-pressure cold plasma processing technologies, including corona, dielectric barrier discharge (DBD) and glow discharge-based processes. He then describes in detail some of the most important basic

manufacturability needs for industrially feasible applications of plasma technology. These include cool processing, large substrate area processing and high throughput. He states that the atmospheric pressure plasma (APP) type intrinsically capable of meeting the size and temperature constraints needed for textile processing is the atmospheric pressure glow discharge (APGD). He then describes, in detail, some existing APP equipments for textile processing. The APP equipment configurations for textile processing can be categorised according to the generic treatments that they can run and hence the kinds of surfaces that they can deliver. There are four main treatments: air treatment, controlled atmosphere treatment, gas precursor coating and liquid precursor coating.

According to Herbert, a key distinction between the different APP options is how they perform in relation to the critical ratio of added value/cost. Added value is strongly related to surface functionality or performance: the more sophisticated and powerful the properties of the treated surface, generally the higher its value. The author states that there is no 'superior' APP technology and that in the current state of development of plasmas for textile manufacturing there is no single ideal and clearly superior plasma technology for this. He adds that atmospheric-pressure plasma is not intrinsically superior to low-pressure plasma or *vice versa*.

In Chapter 5, Thomas Stegmaier, Alexander Rau, Albrecht Dinkelmann and Volkmar von Arnim describe atmospheric plasma systems with specific reference to the corona and DBD treatments of textiles for technical applications. The reasons why plasma processes at atmospheric pressure are advantageous for the textile industry are discussed, followed by a general introduction to the corona and DBD systems. The authors then describe some special adaptations of corona and DBD technology to textile processing, including the principles of the processes and equipment, mechanisms of surface activations involved, application of DBD treatment in some types of technical textiles and the stability of the effects after DBD treatments. Corona systems in combination with aerosols are described and examples are given of some hydrophobic and oleophobic treatments of textiles using the DBD type of plasma technique in combination with gas polymerisation and vaporised liquors.

In Chapter 6, Dirk Hegemann and Dawn Balazs have written about the nano-scale treatment of textiles using plasma technology. Plasma technology can be used for ablation and deposition processes. While ablation enables a complete cleaning of textiles from manufacturing residuals, deposition can be controlled in the nanometer range to achieve new functionalities. The textile properties remain unaffected by the treatments, both being dry and eco-friendly. Some advantages of plasma technology for the textile industry are described looking specifically at the advantages of nano-sized