"NEW-OLD" TEXTILES BY NANOTECHNOLOGIES: BUSINESS OPPORTUNITIES FOR ADVANCED ECONOMIES

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Abstract

Nanotechnology (NT), revolutionising the world of advanced materials, is fast becoming an important busi-ness area for many companies in different range of industries. Among these, textile manufacturing seems to be actually the most thriving and promising sector offering high-specialized and performing materials.

NTs could represent for European and US textile operators, in a globalized scenario, an excellent opportunity to develop next-generation textiles and to provide enhanced properties and new added-values to standardised and, by now, very cheap textile imported products, revitalizing, in this way, an old hi-labour sector in advanced post-industrial economies, no longer capable of price competition wars against lower-wage countries of Asia and Eastern Europe.

In this paper, we'll provide a comprehensive assessment of some of the most important NT textile finish-ing applications.

We'll deliberately exclude the textile nanofibres production technologies and the newer appli-cations of NTs for "smart & intelligent" textiles, postponing their review to future papers.

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Riassunto

Le nanotecnologie, rivoluzionando profondamente e repentinamente il mondo dei materiali avanzati, stanno sempre più diventando un'importante opportunità di guadagno per molte industrie in differenti settori di attività. Tra questi, quello tessile sembra essere quello più promettente dal momento che già da qualche anno offre pro-dotti altamente specializzati e all'avanguardia.

Le nanotecnologie applicate al tessile potrebbero rappresentare, specie per i paesi avanzati, un'eccellente opportunità per contrastare l'inarrestabile "inondazione" di prodotti tes-sili a basso costo e a basso valore aggiunto provenienti dai paesi emergenti, risollevando, in tal modo, un settore che è in crisi profonda da ormai diversi anni.

In questa nota, offriremo una panoramica delle più importanti applicazioni delle nanotecnologie a fibre e tessuti, finalizzate a migliorare, per mezzo di trattamenti di finitura su-perficiale, le loro caratteristiche e proprietà funzionali.

L'esame delle tecnologie di produzione delle cosiddette "nano-fibre" e dei tessuti "intelligenti" sarà trattato in note successive.

Keywords: textile finishing, nanotechnology, fibers, functional properties.

Introduction

Nanotechnology (NT) is nowadays considered a fundamental strategic knowledge tool and one of the most promising technologies and economic drivers of the XXI century. However, there is not a valid internationally definition and terminology about NT; in fact, ISO/TS 80004-1 standard, "Nanotechnologies - Vocabulary - Part 1: Core Terms", which has been approved by ISO Working Group TC 229 and lists a number terms related to nanotechnologies and nanomaterials, has not yet been published.

According to the National Nanotechnology Initiative (NNI), NT is «the understanding and control of matter at dimensions between approximately 1 and 100 nanometers, where unique phe-nomena enable novel applications.

Encompassing nanoscale science, engineering, and technology, NT involves imaging, measuring, modelling, and manipulating matter at this length scale» (see http://www.nano.gov/html/facts/whatIsNano.html).

It is a nanostructure whatever presents at least one dimension of nanometre size for the construction of materials, devices or systems with novel or sig-nificantly improved properties due to their nano-size (1).

Nevertheless, because NT has still limited relevance in consumer-oriented applications, the tex-tile and clothing sector, despite being considered a traditional area, is nowadays one of the most recent pioneers in introducing NT in their products and having huge economic potential for the industry's growth in the short-time.

NTs, in fact, permit – manipulating individual atoms, molecules and bulk matter (the so called "molecular manufacturing") – to engineer new nanofibres and a relatively easy modification of the physical, chemical and functional properties and performances of the textile mate-rials (fibres, yarns, fabrics).

Some of these new improved and innovative characteristics for "old" ma-terials, like natural fibres e.g., are: tensile strength, self-cleaning, UV-protection, high durability, water repellency, fire retardancy, antibacterial effects, and so on.

These features allow textiles to become *multifunctional*, saving on resources, without compromising their inherent favourable properties, including processability, flexibility, washability and softness.

Even if, only few of these applications have already reached the market maturity (with a con-sequent low penetration: less of than 1% of the textile market) (2), actual primary objective of the ma-jor European and US apparel companies and of all operators and stakeholders involved in textile trade and industry, that continuously invest in research and implementation of new technologies, processes, products and fabrics, is to provide new added-values to standardised and, by now, very cheap textile imported products, that have forced many small and medium enterprises (SMEs) to close and thousands workers to lose their jobs.

NTs, in fact, could represent, in a globalized scenario, an excellent opportu-nity to develop next-generation technical fabrics and to revitalize an old hi-labour sector in advanced post-industrial economies, no longer capable of price competition wars against lower-wage countries of Asia and Eastern Europe.

Nano-finishing technologies offer the exciting opportunity of mixing the better qualities of natural and synthetic fibers, producing in this way advanced fabrics that can easily meet and satisfy the consumers' need and demand. Nanoparticles (NPs), such as metal oxides (e.g., TiO_2 and MgO) and ceramics, present better affinity for fabrics and a longer durability of their functions compared with conventional methods, which, adopted till now to give improved properties to fabrics in finishing processes, do not lead to permanent effects and will rapidly lose their functions after wearing or do-mestic laundering (3, 4).

A largely adopted method to apply NPs onto textiles is coating, through different techniques: spraying, transfer printing, washing, rinsing and padding (at a suitable pressure and speed), that is the most commonly used, followed by drying and curing.

Other processes to obtain NT products are Sol-gel process, Layer-by-layer deposition, Physical Vapour Deposition (PVD), Chemical Vapour Deposi-tion (CVD), Atomic Layer Deposition (ALD) (5, 6).

The aim of all these processes is to maximize the nano-structured surface in order to increase the efficiency of the stressed power through a considerable augment of the number of active particles per unit area, which can reach virtually a 100% covering.

NPs, in fact, present a large surface area-to-volume ratio, thus vastly improving their bactericidal and fungicidal effectiveness (7).

In this paper, we'll provide a comprehensive assessment of NT finishing methods and materi-als for textiles, by a review of their best improved properties: water repellence, self-cleaning, anti-microbial effect, UV-protection, fire-retardancy, anti-static feature, moisture dissipation, colourability and wrinkle resistance.

We'll deliberately exclude the textile "nanofibres" formation technologies and the future development of NTs for "smart & intelligent" textiles, postponing their treatments to future papers.

Water repellence

Hydrophobic finishing to textile surface can be obtained by creating a rough structure or using materials with low surface free energy. Any surface is hydrophobic when its contact angle (θ) is above 90°.

Available technologies to obtain water repellent textiles are coating technology and plasma treat-ment. By the first process, nanoparticles are added to textiles during the finishing bath/process. This technology is the most widely used at this moment (e.g., Nanosphere-Schoeller, Nano-Tex, MincorTX-BASF).

By plasma technology, water repellence is achieved through a surface treatment using plasma. Despite few products on the market making use of this technology it is still under development (2). Fabrics are made in a way to be very similar, replicating surface structure and texture, to the lotus leaves, having, on their surface, micro-buds of 10-20 microns height and a contact angle of about 142°. In this way, the water and oil droplets – and the bacteria, stain and dirt particles carried away with them – not having a significant contact surface with these coated textiles, slide-off very easily, keeping the surface always clean (*lotus effect*, discovered, for the first time, in 1982 by German bota-nist Willhelm Barthlott).

Apart from Nano-Tex, to obtain the same effect, Schoeller (http://www.schoeller-textiles.com), a Swiss-based industry, developed the NanoSphere™ finish treatment, that use gel-forming additives to impregnate the structure of the fabric, obtaining, in con-junction with Soft-Shell®, another proprietary technology, an hydro-phobicity effect thanks to the roughness and texture of material surface, without affecting its softness and abrasion resistance (three-dimensional surface structure) (8). As a result, during a shower or rain, water droplets bead up and roll off and surfaces stay dry and clean.

Self-cleaning

To get an optimal self-cleaning ability it is necessary a *super*- or *ultra-hydrophobic* treatment with a very high water contact angle (above 160°) and a very low roll-off angle, increasing, in this way, surface roughness with a larger geometric area and easily removing particles of all kind (dirt, stain, oil, mud etc.) adhering to fabric surface.

Self-cleaning textiles could find wide use in medical and sporting applications. To obtain a self-cleaning property, it is possible to coat the textile materials with metallic ions or metallic com-pounds, like TiO_2 or ZnO (with different band gaps, respectively, 3.2 and 3.37 eV), that react with or-ganic matters (dirt, stain, bacteria, pathogenic fungi), obtaining a sterilising effect and the discolora-tion of stains (9).

Metal oxides, in fact, such as TiO_2 ZnO, MgO and CaO are very interesting because they re-sult unstable and aggressive against microorganisms under specific environmental conditions but, at the same time, generally safe to human beings and animals (10).

In particular, *titanium dioxide NPs*, mostly produced by sol-gel processing method (11), are very interesting especially for their photo-catalytic activities. In fact, their molecules, activated by an energy of UV-light higher (with $\lambda \leq 387.5$ nm in anatase form) (12) than its band gap, emit an electron (e-) that, jumping from the valence band to the conduction band and combining itself with O_2, in the air or water, forms active O_2- (*photo-oxidation*), while the electron hole (h+), reacting with water, generates hydroxyl radicals (OH-). O_2- and OH- catalytically dissolve organic compounds (odour mole-cules, bacteria and viruses), respectively, into CO_2 and H_2O (*photo-catalytic effect* by inverse *redox* reaction) (13, 14).

TiO_2 NMs have unique properties such as higher stability, long lasting, safe and broad-spectrum anti-biosis (15) and are applicable in many fields such as self-cleaning, anti-bacterial agent, UV-protecting agent, environmental purification, water and air purifier, gas sensors, and high efficient solar cell (14).

To enlarge the light absorption range of TiO_2 from UV to visible spectrum and notable increas-ing the efficiency of photo-catalytic activities, in many applications it has been investigated the addi-tion of noble metals, such as Au or Ag, forming *metallic TiO_2 nano-composites* (16, 17), or the reac-tion with hydroxyl propyl cellulose (HPC), forming *non-metallic TiO_2 nano-composites* (18).

Zinc oxide nano-composites – prepared by wet chemical method (1), using $Zn(NO_3)_2$ and NaOH, as precursors, and soluble starch as stabilizing agent – present some excellent properties like exceptional mechanical strength, antistatic, antibacterial (reaching maximum reduction rates upper to 85% for *E. coli* and near to 95% for *S. aureus*) (10), a lower cost compared to silver NPs, white appear-ance and UV-blocking properties (9).

Another type of self-cleaning finishing treatment is produced by polytetrafluoroethylene (PTFE), that, in nano-scale, prevents the formation of bonding forces between a stain particle and the textile surface.

This technology, formulated for resin-treated on cottons and synthetics fibres, balances the body temperature and allows the breathability of the fabric (5).

Processes, adoptable to obtain a self-cleaning surface, could be: sol-gel treatment, plasma technology, electro-hydrodynamics (EHD) method, photo-catalysis. Sol-gel is formed by the hydroly-sis, and successive condensation, of a colloidal suspension of solid NPs (sol) and solvent (gel).

By plasma treatment, some chemicals, like as fluorocarbons and silanes/siloxanes, are polymerized and deposited, by a coating process, on textile surface as nano-particulate hydrophobic film, creating, by nano-whiskers, *a peach fuzz effect*. Hydrophobic fluorocarbons ($\theta \cong 120°$) are organic chemicals con-taining perfluoroalkyl residue, in which hydrogen atom have been replaced by fluorine, and presenting high thermal stability and low reactivity (19).

EHD can use a small mouthpiece, high-voltage charged, through which passes a charged ray of a diluted solution of PS/DMF (polystyrene/N,N-dimethylformamide) that forms, solidifying after the evaporation of the solvent, NPs intermixed with nanofibres. These PS materials, having $\theta>160°$, consti-tute a super-hydrophobic finish and a self-cleaning film.

Antimicrobial effect

Under proper temperature and humidity conditions, in contact with human body, textiles, es-pecially those made by natural fibres, will become a good media for proliferation of microorganisms, which can be resulted in damages, skin irritations and infections.

For these reasons and for increasing public concern about hygiene, recently, there are many investigations about antibacterial mech-anisms modifications of textiles using NTs.

Antimicrobial property, but, very often, without self-cleaning effect, can be achieved by a con-trolled fibres release of embedded biocide agents like as triclosan, Cu^{2+}, Ag^{+}, and so on.

For example, nano-sized silver, releasing Ag^{+} ions, presents opti-mal activity (reaching bacterial reduction rates near to 100% even after several home launderings), for a long time, against a broad range of germs (bacteria, virus, parasites etc.) even in concentrations as low as 0.0003÷0.0005% (so it has been defined an "oligo-dynamic" molecule), non-toxic for higher life forms and not-expensive at all.

In fact, nano-silver, being extremely reactive with proteins, in par-ticular bonding with thiol (sulfydryl, –SH) groups of cellular enzymes, and interfering with the transfer system of the microbial cell membrane, inhib-it cellular metabolism and growth of fungi and bacteria and microorganism dies (20). Antimicrobial ac-tivity of silver particles depends by their dimensions: smaller particles, greater antimicrobial effect. Nano-silver par-ticles are generally smaller than 100 nm and contain 20-15,000 silver atoms (21).

Various methods have been adopted for the preparation – using dif-ferent reducing agents, like as sodium borohydride ($NaBH_4$), formalde-hyde, sodium citrate, hydrazine, ascorbic acid, glucose and γ-rays or UV irradiation in various colloidal solution, like as *sulphur nano-silver ethanol based colloid* (SNSE) (20) – of silver NPs: photo-catalytic reduction, chemical reduction process, photo-chemical or radiation–chemical reduction, metallic wire explosion, sonochemical, polyols, matrix chemistry, photo-reduction, reverse micelle-based methods and even biological synthesized (14).

Nano-sized silver, titanium dioxide and zinc oxides colloidal solutions (25-50 ppm) can treat various textile fibres, both natural that synthetic ones.

In particular, nano-structured materials, like polypropylene/silver (PP/Ag) nano-composite fi-bres, can be prepared, by co-extrusion spinning process, to give an optimum permanent antibacterial activity, evaluated to be effective according to AATCC test method, to common synthetic textiles (22).

By the way, PP, the most used synthetic fibres – because cheaper, stronger and they do not contain any polar groups in their backbone even if they are more subjected to microorganism attack because they comprise methyl chains – could have more useful applications, especially for sanitary purposes (fil-ters, diapers, surgical masks, etc.) (23).

Other nano-structured anti-microbial agents include titania nanotubes (TNTs) (24), gold or copper NPs, carbon nanotubes (CNTs) (25), nano-clay and its modified forms, agents based on gallium composites (Ga_2O_3) (26), and inorganic nano-structured loaded organic carriers: liposomes, den-drimers, nano-capsules and cyclo-dextrins or CDs (characterized by a hydrophobic interior and hydro-philic exterior layer) (6, 14). However, anti-bacterial activities of many of these NPs, except Au com-pounds, seem to be remarkably less than that silver ones.

A trouble of self-cleaning and antimicrobial textile treatments is that sometimes the active substrates also destroy unwanted organic matter of fibres and coating materials. There is the need, then, to use a special protective buffer layer.

UV-protection

Very numerous and different, each other, are the applications of the NMs on fabric to block UV-light by integrating fibres with metal particles, dyes or pigments, generally, with a conventional finishing method of dip-pad-dry-cure (e.g., for ZnO) or sol-gel techniques (e.g., for TiO_2 and SiO_2).

Nano-inorganic UV-blockers, like some semiconductor oxides (TiO_2, ZnO, SiO_2, Al_2O_3), pre-sent, more than organic and conventional size blockers, superior properties: no-toxicity, superior chemical stability at high temperature exposure, better durability, an exceptional *UV-protective factor* (UPF) rating (weighting UV-A and UV-B radiations) and upper ability to absorb and scatter UV radiation (13). In fact, very small particles, like oxide nanorods (10÷50 nm in leght), have an higher blocking efficiency, because, according to Rayleigh's scattering theory, the intensity of the scattered light varies as the sixth power of the particle size and varies inversely with the fourth power of wavelength (27).

The sol-gel method is the UV-blocking most used treatment for cotton, assuring to maintain the effect even after 50 home launderings. Fabrics coated by methyl red dye with nano TiO_2, synthesized by polyol method, present good antistatic and antiflammable properties too (28). TiO_2 is also used for air freshening, removing bad odours from confined space (29).

UV visible spectrum blocked by ZnO NPs synthesized with 0.5% soluble starch (amylose com-ponent of starch) shows peak absorption at 361 nm. By using effective mass approximation, the size (diameter) was calculated to be 40 nm (1, 9).

Some authors suggest the use of natural wool short fibres, generally wasted during processing, pulverising them into nano-scale particles and so treating pure cotton fabrics to obtain additional func-tions such as UV protection, IR absorption and warmth retention. Analogue use can be done of silk, cotton or hemp (30).

Fire-retardancy

To increase fire or flame retardancy (FR) of a fabric and, at the same time, its strength, anti-bacterial and sterilizing efficiency (31), UV-light and chemical protection, some investigations are concerned with *clay NPs* or *nanoflakes* (in particular, *montmorillonite*), composed by different types of hydrous aluminasilicates, or other nano-matters, such as diamine (diaminodiphenyl methane), boroxosiloxanes or Sb_2O_3 (32, 33). With nano-clay flakes the heat distortion temperature (HDT) in-creases from 65°C to 152°C (34). One of the major obstacle of these processes is to set up the disper-sion of the nanosized fillers into the polymer matrix compounds, trying to avoid their natural interac-tion tendency, that heavily obstacle the successive aggregation process with yarns.

MgO, Al_2O_3 and SiO_2 can also increase the mechanical strength, abrasion resistance and the fire retardancy of textile.

Other NMs used as additives in extrusion, coating and finishing textile applications (composite fibres) to enhance FR when incorporated into a ethylene-vinyl acetate (EVA) polymer-matrix, are car-bon nanotubes (CNTs) (35), which, discovered in 1991 by the Japanese scientist Sumio Iijima while working with NEC (36), could be constituted by different wall layers: single walled (SWCNT), double walled (DWCNT) or multi walled (MWCNT) carbon nanotubes that consist of several (usually 7 to 20) concentric cylinders of SWCNT. All of them are mainly manufactured by electro-spinning process (37).

They can reach lengths of several millimetres and present extraordinary characteristics: very high elec-trical and thermal conductivity (38), Young's modulus in the terapascal (TPa) range, considerable ten-sile strength (~200 GPa), equal to 100 times stronger than steel, having 1/6th of its weight, elastic strain up to 5% and breaking strain of 20% (14, 32).

The most used production method to integrating the described anti-flame nano-composites into fabrics are melt blending or melt spinning processes (39).

Anti-static feature and moisture dissipation

Man-made fibres (nylon, polyester), as it is well-known, absorbing negligible quantity of H2O, have low moisture content and accumulate on them static charges, therefore acting as isolators. Elec-trically conductive NPs of TiO_2, ZnO whiskers, Sb-doped SnO (antimony-tin oxide or ATO), SiH_4 nanosol (40) and polytetrafluoroethylene (PTFE or Dupont's Teflon) (41), creating bound between hy-droxyl groups and water and dissipating the accumulated electrical charges, have been successfully used to make anti-static synthetic fabrics, in a much more efficient way than conventional techniques, which present a low laundry-proof properties.

For example, it is possible to "exploit" the "electric holes" of the semi-conductive TiO_2 va-lence electrons, lifted into an excited state. In fact, light-generated electron-holes can migrate to the surface, combining themselves with atmospheric oxygen, to give, as seen above, superoxide radicals, which quickly attack the nearby organic molecules or can oxidize lattice oxygen at the surface of the material, leaving oxygen vacancies which can be filled by adsorbed water (super-hydrophilicity effect) (42). By layer by layer, then, it could obtain a hydrophilic polyethylene fibres alternating thin film of TiO_2 with polydimethyldiallylammonium chloride (PDAC) (39, 43).

Such antistatic “nano-treatment”, in conjunction with high electrical conductivity and excel-lent physic and mechanical properties (high tensile strength and conductivity, low electrical resistivity, high thermal stability, toughness, etc.), can also be produced on synthetic materials by increasing the conductivity of the fabrics, using, e.g., carbon NPs (CNTs or CB) (44), special polyacrylonitrile (PAN) fibres (32) or by conducting electro-active polymers (CEP), like polyaniline, polypyrrole, polythio-phene, and derivatives; all of them included into the polymer matrix and subsequent electrospinning (39). These special composite fibres are created by chemical oxidative deposition technology (32).

Colourability and wrinkle resistance

Hydrophilic surface modifications, e.g., by nano-thin coatings (e.g., through plasma polymeri-zation of C_2H_2 mixed with ammonia), can also improve the dyeing properties of fabrics. Besides, nano-particulate pigments, like as carbon black (CB), can directly be used as, quite permanent, dyes. CB, in fact, can be modified to obtain carboxylic surface structures and facilitate the absorption by tex-tile fibres (polyamide, cotton and acrylic ones) through exhaustion process (45).

Anti-wrinkle function can be obtained, besides the use of resins – that present different prob-lems, like low tensile strength, abrasion resistance – as conventional method, by nano-titanium dioxide (applied on cellulose with a carboxylic acid as catalyst) and nano-silica with $C_2H_2(CO)_2O$ (maleic an-hydride) for cotton and silk respectively (46).

Nano-Care® fabric protection, the upper mentioned Nano-Tex technology, allow to obtain ease of care and superior stain repellency and wrinkle resistance by molecularly changing the fabric.

Working on the nano-scale and permanently attaching to the fibres, the chemistry provides greater du-rability than repellent coatings at the same time as permitting to the fabric to remain breathable, soft and hand feel.

Conclusions

There is no doubt that in the next few years NT, overcoming the limitations of traditional tech-niques and enhancing the processes used to add desirable attributes and special functional properties to textiles, will become a very profitable business area. However, before industrial mass production and large commercialization of nano-products, there are some heavy aspects to evaluate.

First of all, the possible harmfulness of uncontrolled release of NPs, because of their similarity to asbestos fibres, on human health, especially, of workers and consumers. Even if the existing bio-logical and epidemiological researches seem to indicate that this impact is quite limited, current stud-ies on environmental, health and safety (EHS) concerns are very few.

Another question is the issue of manufacturing costs that are very high and represent the most important real obstacle towards a mass production of NP. In many cases, the issue of high production costs make mass production and up-scaling processes less viable.

In fact, the costs – related to the equipment investments, to the hiring of expertise, to the purchase of raw materials, etc. – are not yet compensated by the NT added-value of nano-finishing treatments, especially if compared with conven-tional ones. For these reasons, only few nano-applications have already reached the market maturity and their penetration is still very little.

Other barriers to the penetration of NT into textile industry are:
1) textile operators are many fragmented and hesitant to accept novelties;
2) among them, the knowledge about benefits and risks of NT is very poor;
3) lack of qualified experienced personnel and specialized machineries;
4) for many processes, the reliability is still very low;
5) several other technical limitations (commercial availabil-ity of nano-materials, complexity to get the preferred particle pre-dispersion formula-tion, etc.);
6) lack of a clear and effective legislation and regulation on healthcare, since exiting laws are inadequate;
7) it is necessary, then, to appoint scientific test methods to determine whether a product incorporating na-nomaterials, giving adequate and real information to consumers in order to protect their interests.

It is necessary a pressing international standardization – on the same bases of the chemical substances classification in REACH and GHS

(47) – of taxonomy and terminology of NMs, analytically determining their physical and chemical properties in order to facilitate the comparability of scientific researches.

Considering the enormous role of textiles in human life and the increasing use of NT in this field, there is the imperative need of multi- and interdisciplinary research collaborations, especially to deal with the pressing question of the impacts of uncontrolled release of NPs and to define, trough risk assessment and risk management exposure analysis, effective regulatory schemes and voluntary meas-ures without constraining the sector growth.

At this point of view, the scholars of Commodity Science could make their significant contribution for a considerable advancements of NP in textiles enhancing R&D investigations and industrial scale economic simulations.

Commodity Sciences are also important to summarize, critique and communicate technical results, providing an accurate, indispensable base of information and safety evaluation for public, consumers and all decision-makers.

For example, it seems important to communicate, by adequate labeling systems, to consumers about the possible toxic effects of some NMs used on products, after having determined, through re-search studies on exposure-response on animals and humans, their "non-observed adverse effects lev-el" (NOAEL) (48).

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