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Characterization of nanocomposite coatings on textiles: a brief review on Microscopic technology

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There has been an increasing consideration in nanotechnology during the present decade due to its enormous potential in applying and creating novel materials for enhanced properties and applications. Numerous studies were undertaken in improving the textiles and clothing properties and performances by applying nanocomposites. Microscopy technique which is a fundamental tool in nanotechnology has been widely employed for the investigation of particle size, size distribution and the homogeneity of nanocomposite coatings. This technique can also be used to investigate the properties of surface, thickness of applied nano layer, and/or 3D morphology of the surfaces. Current microscopy methods contain a vast majority of analysis that can be applied to characterize nanocomposite coatings on the textiles. These analyses includes scanning electron microscope, transmission electron microscope, atomic force microscope, scanning probe microscope and laser scanning confocal microscope. In this chapter, approaches to develop nanocomposite coating on textile materials are summarized and microscopy methods and analysis conducted by researchers to identify and determine the surface properties of nanocomposite coatings on the textile fibers are discussed.

Keywords: nanocomposites; textile; microscopic techniques

1. Introduction:
Nanotechnology has been involved in textile performances improvement and/or new functions for several years [2] and has been caught enormous attention in the textile field [2]. Nanomaterials are ultrafine materials in at least one dimension to the size of nanometer order (below 100nm) [1]. They can be applied to textiles to modify or enhance its properties. Recent studies are mainly focused on applying nanomaterials on producing nanostructures during manufacturing, finishing and coating process [2-6]. The application of nanotechnology in textile produced multifunctional or special functions, such as antibacterial [7], superhydrophobic [8], and fire retardant [9] products. In conventional methods, such as dip coating, nano colloidal has been used in coating process [10], while the wide ranges of coating methods have been promoted [11-13].

In nanotechnology, the primary issue for researchers is to examine the nano scale material so a variety of microscope approaches have been developed. In this chapter, the most widely used nanocoating process is summarized and the application of microscope analysis including electron microscopy, scanning probe microscopy (SPM), and laser scanning confocal microscope (LSCM) in textiles is discussed.

2. Methods for generation of nanocoatings on textiles:

2.1 Sol gels technique:
Colloidal solutions contain typically metal oxide nanoparticles in either aqueous or organic solvents which constitute nano sols. The sol-gel process is one of the most widely used methods in material science [14, 15]. It is a wet process that is broadly employed in the textile field using a simple pad or dip coating [16, 17]. Nano Sol-gel method has been applied for preparing self cleaning [18], antibacterial [19], and water repellent finishes [20] on fabrics.

Anatase titanium dioxide nano crystals were prepared by sol-gel method. In this procedure, tetraisopropl orthotitanate was added to deionized water containing acetic acid at the room temperature and the aged sample was used for coating the cotton fabric by a dip-pad-dry-cure process. It was shown that the treated fabric had a remarkable photo catalytic self cleaning performance and it had ability to decompose a colorant and degrade red wine and coffee stains as well as proper protection against UV radiation [18]. In another study, tetrabutyl orthotitanate, used as precursor, was added into a mixture of citric acid (CA) and ethanol, then after a certain period of time, a solution containing a mixture hydrochloric acid in deionized water was dripped during agitation. Ultimately, transparent sol was achieved. This sol, containing nano TiO2 sols, triethanolamine (TEA) and sodium hypophosphite (SHP), was used for applying cotton fabric in two-dip-two pad coating. The finished fabric has been suggested for UV resistance and anti wrinkle applications [21]. In another attempt, titanium oxide was prepared mixed with silver nanoparticles using a sol-gel route. In this procedure, first of all a polymeric sol (PS) was formed by mixing tetraisopropl orthotitanate (TIPT) with deionized water, hydrochloric acid (HCL), and ethanol as a solvent. The aged sol after 2 days was diluted in an excess
of deionized water and then autoclaved. At this stage, after water removed, the sol diluted with ethanol to form a crystalline suspension (CS). Then silver nitrate was poured in ethanol and added to CS solution and the mixture was stirred and then exposed to UV radiation. The product, mixed Ag-TiO$_2$ liquid suspension, was applied for impregnation textile fabric. The functionalized textile exhibited a strong antibacterial activity [22].

Superhydrophobic fabric with silica nanoparticles was achieved by sol-gel process. To prepare a spherical silica nanoparticle, Tetraethoxysilane (TEOS) was added to the mixture of distilled water, ethanol and ammonium hydroxide. Ammonium hydroxide acts as a catalyst. The textile fabric were treated in the sol of silica nanoparticles and cured. The fabric treated showed the water repellent property and the contact angle of 130° was achieved [23]. Moreover, coating a complex layer of silica nanoparticles and perfluorooctylated quaternary ammonium silane coupling agent (PFSC) was used to prepare the super hydrophobic fabric. In this procedure, silica sol was prepared by alkaline hydrolysis of TEOS in a mixture of ethanol, water and NH$_3$. Then the fabric was immersed in the silica sol and padded with 2 dips and 2 nips. After drying, the fabric was immersed in the methanol solution contains PFSC, and then padded with 2 dips and 2 nips and cured at 160 °C. The treated fabric showed a high hydrophobicity and oleophobicity and the water and oil contact angles of about 145° and 131° were achieved, respectively [24]. In another study, sol-gel coating of TiO$_2$ was applied to prepare a superhydrophobic fabric. TiO$_2$ sol (solution A) was prepared by adding Tetraethyl titanate into the anhydrous ethanol during stirring. Then the solution B, containing deionized water, acetic acid and anhydrous ethanol, was added to solution A. The aged solution in ethanol solution was used for coating cotton fabric by pad process. The treated fabric posses a contact angle larger than 150° with water and also provides a good UV protection [25].

2.2 Magnetron Sputter Coating:

Nanocoatings by physical treatments methods have attracted a great deal of attention because of the environmentally friendly technology. Sputter coating is one of the physical vapor deposition (PVD) methods. Thin film sputtering fabrication has become a common manufacturing process for a variety of industries and sputter coating has been used to deposit various materials on the textile fabrics. It has been found that a quadruple magnetic field increases the deposition rate, so magnetron sputter systems are widely used for thin film deposition. Magnetron sputter coating is a vacuum process that applies a high voltage across a low-pressure gas to create reactive gas molecules. During sputter coating, reactive gas molecules strike a target and cause atoms from the target to travel to the substrate [26-28].

Metals and metal oxides can be deposited on the textile fabrics using magnetron sputter coating. For example, zinc oxide (ZnO) [29-30] and copper (Cu) [31] nanostructure were deposited on the nonwoven fabrics by sputter coating. Magnetron sputter coating has also been used for depositing nano structure thin film on the surface of nonwovens. In the sputter coating process, time has important impact on the grain sizes of the particles and the surface smoothness. Sputtering silver metal on the polypropylene nonwoven fabric has been investigated. In this procedure, silver nanoparticles were formed when the sputtered film had a thickness of less than 3 nm and with increasing the sputter time an increase of the grain size and a more compact film was observed. The metalized fabric had antibacterial properties which were improved with increasing the sputtering time. Moreover, when the film thickness was increased over 50nm, the sputtering time led to the more compact film and resulting to improve the electric conductivity [32].

Nano size Fe$_2$O$_3$ is coated on the surface of polyamide 6 (PA 6) nanofibers and it was shown Fe$_2$O$_3$ improved the thermal stability of the composite nanofiber [33]. In another attempt, a transparent nanofilm was deposited on the PA 6 nanofibers by tin-doped indium oxide (ITO). ITO deposition led to remarkable improves in the electrical properties of the PA 6 nanofibers. Increasing the ITO thickness caused an improvement of the coverage of ITO clusters which in turn resulted to a better conductivity [34]. In another study, propylene (PP) fabric was coated with ITO and aluminum doped zinc oxide (AZO) separately and their physical properties were compared. It has been shown that PP deposited with ITO and AZO have different properties. The AZO coating provided better UV shielding effect in comparison to the ITO coating at the same thickness fabric deposited with ITO showed a better surface conductivity which in the same thickness [35]. The polymer-metal nanocomposite film prepared by the cosputtering of gold and polytetrafluoroethylene (PTFE) from two independent magnetron sources was conducted. It was elucidated that the electrical and optical properties of the product depends on the microstructure parameters [36].

2.3 Plasma:

Plasma is a dynamic mix of ions, electrons, neutrons, photons, free radicals, Meta stable excited species and molecular and polymer fragments. Plasma can be achieved in different atmospheric conditions, either low pressure or atmospheric pressure, also can be achieved in different types of power including low-frequency, radio-frequency, or Microwave [37]. The plasma technology is widely applied in textile and polymer industries for a wide range of application such as shrink-resistance [38], anti-scratch [39], superhydrophobic [39], superhydrophilic [39], and flash-fire resistance [40]. It is a suitable technique for modifying the structure and topography of the surface as well as depositing of nanocomposites into the surfaces [38-40].

Furthermore plasma methods including radio frequency plasma (RF-plasma) and microwave plasma (MW-plasma) have been applied as a pre-treatment to improve deposition process, and then the loading of TiO$_2$ on the textile surface is done by wet chemical technique that leads to form a controllable and uniform modification of the textile surfaces with
increasing the polarity and hydrophilicity of the textile surface. These modified coated textiles show a remarkable photo-oxidative activity under the visible light [41].

Nanocoatings with water repellency property were fabricated using plasma [42, 43]. Hydrophobic textile is achieved by atmospheric air plasma containing aerosol and injecting fluorocarbon directly into the plasma dielectric barrier discharge. In this process, the aerosol atomization of fluorocarbon dispersion is used to promote the chemical grafting. This is a useful chemical grafting method. It was shown that an atmospheric plasma treatment is not adequate to crosslink the nano fluorocarbon coating so to increase the degree of cross-linking the treated fabric must undergo a thermal curing at 180 °C [43].

Deposition of silver nanostructure on the fabric surface embedded within a plasma-polymer matrix was done. In this process, polymerization of a mixture of acetylene and ammonia (C2H2/NH3) while using RF discharges was applied. To achieve a nonporous cross-linked network deposition during plasma polymerization, co-sputtering of a silver target with Argon was used to prepare in situ embedded nanoparticles on the coating surface. Plasma co-sputtering enabled the formation of multi functional surfaces. It was also elucidated that coating was permanent and useful for antibacterial applications [44].

2.4 Layer-by-layer (LBL) technique:

Layer-by-layer (LBL) technique is another method for fabricating a thin layer film and is based on the concept of self-assembled nano layers. LBL process causes to enable modifying multicomposite molecular assemblies with a control on the molecular structure and a high degree of control over the thickness. There is more attraction using electrostatic self-assembly (ESA) because of the simplicity and efficiency. In LBL method, polyelectrolytes with opposite charge were alternately deposited on the fabric surface with wash steps in between. For increasing the thickness, cycles of adsorption can be repeated [2, 45, 46]. LBL technique incorporated to nanomaterials was used for applying a thin nanocomposite on the fabric surface and wide range of functionalities have been imparted to fabric with LBL method [46-48].

In LBL depositions, cationic sited are usually generate on the surface. ZnO nanoparticles were applied on cationized cotton via LBL method. 2,3-epoxy propyl trimethylammonium chloride (EP3MAC) was used and reacted with the hydroxyl groups of cellulose to create cationic charges on the surface of the fabric. In the deposition process, the cationized cotton was dipped into sequential solutions alternately as: (a) anionic ZnO solution, (b) deionized water, (c) cationic ZnO solution, (d) deionized water. The cotton fabric treated with bilayers films showed excellent antibacterial activity and UV protection [49]. In another study, after preparing the cationized cotton fabric by using EP3MAC, it was dipped into the solutions alternately as: (a) anionic Poly(sodium 4-styrene sulfonate) PSS or Nano polyurethane (nano PU) solution, (b) deionized water, (c) anionic TiO2 colloidal solution and (d) the deionized water. For the TiO2/poly(diallyldimethylammonium chloride) PDDA bilayer films, the cationized cotton fabric was dipped into the following solutions alternately as: (a) anionic TiO2 solution, (b) deionized water, (c) cationic PDDA solution, and (d) deionized water. As a result, fabric treated with 16 layers of nano PU/TiO2 and TiO2/PDDA film showed excellent UV protection [50].

The multilayer Al2O3 nanoparticles film on the cationized cotton fabric was fabricated. In this process, the cationized cotton fabric was dipped into the following solutions alternately: (a) the anionic Al2O3 colloidal solution, (b) the deionized water, (c) the cationic Al2O3 colloidal solution, and then (d) the deionized water. The deposition cycle was repeated until 16 bilayers Al2O3. The result showed that the deposited cotton exhibited a better UV protection and a significant flame retardency in comparison to untreated cotton. Silver nanoparticles were deposited on the nickel and the silk fabric using LBL method for antibacterial purpose. The fabrics were separately immersed in the solutions of poly (diallyldimethylammonium chloride) (PDADMAC) and silver nanoparticles capped with poly (methacrylic acid) (PMA) and composed by alternately dipping. As a result, the scanning electron microscope (SEM) images confirmed that the coating on the nylon fabrics was not as uniform as on the silk. After depositing 20 bilayers onto the fabric, an 80% bacteria reduction for the nylon fabric and a 50% bacteria reduction for the silk fabric were observed [52].

2.5 Nanomaterial embedded textile:

Since finishing fabric with nanomaterial is not permanent especially against washing, therefore the nanomaterial has to be fixed into the textile fabric. Three different methods have been suggested to embed nanoparticles on the polymeric matrix. In the first method a cross linking agent is used to entrap nanocomposites. Crosslinkable polymers such as binders have been suggested for this purpose [53-56]. Secondly, an in situ synthesis of nanoparticles on the fabric in order to increase the stability of nanoparticles has been employed as an alternative method of fixation [57]. The other method involves embedding nanoparticles on the fiber polymeric matrix using a carboxylic acid which is a new method with remarkable advantages [58-63].

Cross linkable polysiloxan has been used for stabilizing silver nanoparticles on the textile surface which also has many unique properties such as softness, flexibility and so forth [53-54]. Moreover, using binders to improve the stability of silver nanoparticles [55] and ZnO was elucidated [56].

On the other hand, carboxylic acids have been used for embedding various nanomaterials such as CNTs [58, 59], fumed silica [60, 61], and silver nanoparticles [62]. To prepare CNT embedded textile, CNTs have been absorbed to the
surface using by exhaustion method followed by aftertreatment with BTCA [58, 59]. In another method nano silver and nano fumed silica by BTCA have been applied synchronous on the fabric surface [60-63]. Ultimately, In situ synthesis of silver nanoparticles in boiling point led to stabilized nanoparticles. The synthesis process in the boiling temperature led to the swelling of the fibers and molecular chain and thence it is assumed nanoparticles to form and penetrate into the fabric structure and after the treatment they remained there [57].

3. Microscopic methods for surface characterization of textiles in nanoscale:

3.1 Scanning Electron Microscope (SEM):

Microscopic analyses are essential in nanotechnology. Electron microscopes are one of the most common analysis instruments that use the interaction of emission ray of electrons with the sample atoms to provide magnified image. There are several types of electron microscopes according to the type of electrons that has been using for producing image. Hereon SEM and TEM are two main types of electron microscopes. Electron microscopes are precise instruments and play an important role in nanoscale systems. Electron microscopy can be employed in nanostructure imaging, composition, determine physical properties measurements and even building and manipulating nanostructures [64-67].

SEM scans the surface of the sample with a beam of electrons and the resulting image has a three-dimensional appearance that can be useful for the surface structure investigation while image produced by TEM is rather two-dimensional and the electrons pass through the sample, so the image is not useful for the surface structure investigation [68-71]. The SEM is one of the most important and most widely used instruments in nanotechnology and textile applications. It often requires metal coating on the sample by sputter coating which requires vacuum condition. SEM provides the possibility of directly observing the surface morphology of textile and nanocoatings [72-75].

SEM images can be applied to investigate the surface condition, purity and the structure of uncoated fabric. SEM images were used for comparing the untreated fabrics and nano coated fabrics. Fig. 1a shows that there is no impurity on the surface of untreated cotton fabric. Images in Fig. 1(b,c,d) have illustrated the surface of nano coated fabric with different type of nanoparticles with the same resolution. Fig. 1b confirms that the silver (Ag) nanoparticles were adsorbed homogeneously on the cotton fabric. Achieved SEM images were also applied for particle size distribution and nanoparticles shape determination after in situ synthesis of silver nanoparticles on the surface [62].

Fig. 1c presents the cotton fiber crosslinked with BTCA and hydrophobic silica nanoparticles. It shows the formation of a few aggregated silica nanoparticles with a rough structure that is proper for hydrophobic applications. It is evident that the nanoparticles are relatively well dispersed on the surface with a thin layer of the silica coating [60, 61]. Also Fig. 1d confirms the presence of CNTs and it shows cotton surface after deposition of CNTs by exhaustion method with a few aggregation and homogeneous coating [58, 59].

![SEM images of cotton fabric: a) untreated cotton; b) coated with silver nanoparticles; c) coated with fumed silica; d) coated with carbon nanotubes (at the same magnification) [58, 60, 62]]
SEM was used to investigate the presence of deposited nanolayers and/or incorporate nanoparticles on the surface in the LBL method. Hence, SEM images were applied for a better understanding of the burning behavior of the treated with poly(sodiumphosphate)/poly(allylamine) (PSP/PAAm) as shown in Fig. 2. It was shown that the weave structures of treated fabrics were retained stable and did not observe shrinkage of the cotton fabric after burning. At higher magnification, the surface of the fabric is more distinguished. The untreated fabric has a smooth surface and after 5 bilayers PSP/PAAm the fabric structure remained intact. After increasing the bilayer to 10, some fibers started to link to each other and formed a continuous film, covering the structure of fabric. A close attention to the fabrics after burning revealed the intumescent behavior. The shape and size of the fibers covered with 10 bilayers are similar to those of untreated ones; but any further increase in the number of bilayers coating affect the burning behavior of samples. The surface of fibers were rough that can be due to swelling and expansion of the nanocoating. The SEM images illustrated the intumescent effect of nanocoating which prevents the fabrics to burn [76].

In addition, the SEM surface images of nanocoated cotton fabric by LBL deposition process with poly(sodium-4-styrene sulfonate) (PSS) and poly(allyl amine hydrochloride) (PAH) are illustrated in Fig. 3. It shows the deposition of 13 layers of PSS/PAH on the cotton fabric with and without the use of ultrasonication in the washing step at different magnification. It was clearly shown that the ultrasonication during the intermediate washing step has a great impact and it caused a uniform deposition of bilayers on the fabric. Ultrasonication also caused to remove the excessive deposition of polymers on the entire fabric surface and no crack was observed on the fabric after ultrasonication [77].
In another study, ZnO@SiO2 nanorod array was synthesized on the cotton fabric by a sol-gel method. In this procedure, ZnO seed layer was grown by dipping the fabric in a sol-gel, and then ZnO nanorod array was prepared by a simple hydrothermal process. A SiO2 shell was prepared on the ZnO nanorod array by LBL method. Fig. 4 showed the SEM images of treated and untreated fabric. According to the SEM images, oriented ZnO were densely and uniformly formed on the surface of cotton fibers. The length and diameter of the ZnO nanorods were estimated 2 µm and 440nm, respectively. Fig. 4e showed the top-view image of the SiO2 layer deposited on the ZnO rod array. Also, a rougher SiO2 layer could be observed on ZnO surface. Further analysis with EDX spectrometry confirmed the presence of Si and Zn elements in the LBL-treated nanorod array [78].

3.2 Transmission Electron Microscope (TEM):

TEM is an important technique in textile used in a wide range of applications. It has the ability to provide detailed information about the ultrastructure and it is applied to investigate the internal structure [79, 80]. For example, TEM was used to study the details of the Microcrystalline cellulose (MCC) [80]. TEM provides information on the particle nucleation [81], core–shell structure of the particles [82], crystalline nature [82,83], film thickness [84], particle shape [85], nanofiber diameter [86], distribution of nanoparticle through nanofiber [86] and structure of coating [84].

In many literatures, the shape, distribution, and particle size of nanomaterials were investigated using TEM images. TEM sample preparation for particles that have small dimension can be prepared by deposition a drop of a colloid solution on a grid. Fig. 5 shows silver nanoparticles after synthesis with different methods (thermal, microwave, and ultrasonic) before padding on the cotton surface. Samples were prepared by a drop of dilute solution on a copper grid coated by an amorphous carbon. The images indicated that nanoparticles shapes are spherical irrespective of the method of preparation and the mean sizes of particles are 3-22nm. It was also shown that the silver nanoparticles prepared by
the ultrasonic method were more uniform in comparison to other methods and while in 2 other methods agglomerations of the nanoparticles occurred [87].

In another study, a high resolution TEM was used to investigate the crystallite and the lattice space of a synthesized Dumbbell-shaped ZnO. In this procedure, the ZnO Dumbbell-shaped was synthesized on the cotton surface by suspending the cotton fabric in an aqueous solution of ZnAc₂H₂O and triethenamine (TEA) at ambient temperature. Fig. 6 shows the ZnO nanorod dumbbells had fairly large lattice mismatches at the area indexed using the arrows. Also, the image process by the Fast-Fourier transformation method was done for reducing noise image, as well as the inset in the top right corner showed the power spectrum. The square image which was shown in the top left corner reveals the lattice fringes of 0.23 and 0.2785nm diameter, corresponding to the (001) and (100) planes of ZnO, respectively [88].

TEM was used for observing the cross-section of nanocoated fabric. Fig. 7a,b shows silver nanoparticles loaded on the cotton fabric. The precursor solution was prepared by adding isopropanol to the tollens' reagent and the cotton fabric was dipped in the solution and boiled for 1h. The cotton fabric was imbedded on the epoxy resin and cross-section of fabric was prepared by an ultra-microtome. Fig. 7a illustrates the silver clusters on the cotton textiles and showed penetration of nanoparticles intra-molecular chain by diffusion. Higher magnification of the silver-nanoparticles image was shown in Fig. 7b. Particle size distribution of 2-12nm was observed which indicated highly dispersed silver-crystallites [89].

Fig. 7c,d present the TiO₂-SiO₂ layer on the cotton fabric that is suitable for photo catalysis applications. After deposition the TiO₂-SiO₂ layer, the cotton, fabric was embedded in an epoxy resin and cross-sectioned by an ultra-microtome. The particle sizes were measured 4-8nm and the TiO₂-SiO₂ layer had 25nm thickness. The small crystallites and particle size were beneficial for the photo catalytic applications. Moreover small particles were enough to produce transparent films. In addition, TEM images showed that the TiO₂-SiO₂ layer deposited on cotton had the same structure after 24h an irradiation compared to standard samples and it confirms that TiO₂-SiO₂ layer is stable [90].
3.3 Scanning Probe Microscopy (SPM):

SPM is one type of microscopy that provides images of surface with physical probe of the surface. It was applied to characterize the surface topography and roughness of the surface that are key factors for determining the superhydrophobicity of surfaces [91, 92].

On the other hand, AFM is from family of SPMs with very high-resolution and is a common technique for nanocoatings investigation in textile. It provides access to the surface texture and in contrast to SEM; it does not need any previous coating of samples, so it can form images of samples in a non-vacuum system and also operates in gas or liquid forms. AFM is usually applied to corroborate observations made by SEM and TEM [93-95]. AFM is an effective instrument for investigating the nanostructure of fabrics and also a very high resolution imaging tool. It has been used to view the three-dimensional fibrous structure, surface texture, nanocluster, nanoporous structure and fiber diameters [44, 96-100]. In addition, it was applied to get high magnification image of particles deposited on the fabric. The surface functionalization of fabric by the sputter coating was widely investigated by AFM. It was observed that the interfacial structures are affected by the coating conditions [101-103].

Nanostructures of silver films with different thicknesses were deposited on nonwovens fabrics by magnetron sputter coating for antibacterial and electrical conductivity applications and the silver films were investigated by AFM. The AFM images in Fig. 8 show that the untreated fabric had a smooth surface and with any increase of the sputtering time, the grain size of nanoparticles was increased. In Fig. 8b, there was an evidence of some nanosize particles at the thickness of about 0.5 nm. By increasing the film thickness to 1 nm, the surface was fully covered. In addition, Fig. 8c,d show homogeneous grains of nanoparticles and that the grain sizes was increased. Ultimately when the film thickness reached to 3 nm, the particle size was increased and aggregation with large grain size particle was observed [32].

Fig. 7 A high-resolution TEM image; a,b) the silver nanoparticle synthesized on the cotton fabric; c,d) SiO$_2$/TiO$_2$ deposited on the cotton fabric [89,90]
In Fig. 9 the thicknesses of silver coatings have increased to 50, 100 and 200 nm. Fig. 9 provides an observation of a larger variation in grain size of the particles. In comparison to the thinner film an increase in thickness up to 200nm (Fig. 9a,b,c) cause a more uniform particle distribution on the surface and hence more smooth fabric surface [32].

In another study, AFM was applied to investigate the thin-coat finishing of fabrics that carried out by the sol-gel method. This technique is based on depositing hybrid SiO$_2$/Al$_2$O$_3$ sol. It was synthesized from 2 precursors, (3-lycidoxypropyl)trimethoxy-silane and aluminum isopropoxide on the fabric surface. Nanostructure thin coating film
completely and uniformly covers the cotton surface as shown in Fig. 10. In Fig. 10a, fibrils show on the untreated cotton fabric. On the other hand, bundles of fibrils uniformly covered with sol-gel are shown in Fig. 10b. The AFM images clearly confirmed the change in surface morphology formed by the sol-gel technique. Thickness of fibrils was about 20-30nm so it was assumed that the thickness of the coating could be several tens of nm [104].

3.4 Laser Scanning Confocal Microscope (L SCM):
LSCM is a technique used to obtain high resolution optical images with depth discrimination in three dimensions and has become widely established as a research instrument [105]. Confocal microscopy was applied in various fields in textile including investigating morphology of nanofibers [106, 107], study of dye diffusion in fibers [108], and examination of hairs and fibers [109]. LSCM was applied limited to analyze the nanocoating on the fibers. It was usually used to investigate the nanocoating on the fiber surface coated by LBL method [110, 111]. LSCM was also used to inquire surface of cellulose fiber coated with enzymes, laccase and urea through LBL assembly. In Fig. 11, images of cellulose fiber coated by 3 bilayer of laccase labeled with green fluorescence was shown. LSCM was applied to visualize the location of the laccase multilayer nanocoating. For this purpose, laccase was labeled with fluorescence and assembled with Poly(dimethylallyl ammonium chloride) PDDA. Fig. 11 showed the uniform laccase coating on the surface [112].
In another study, copper and silver based nanostructure coatings on the cellulose were prepared by LBL method. In this procedure, effects of cytotoxicity of coated fabrics were investigated by exposure to mammalian cells. Therefore, LSCM was applied to observe the cells after 48h incubation. Images using LSCM showed healthy cells on both cotton fabric and on fabric coated with Cu nanoparticles, while little/no living cells were observed on the fabric coated with silver nanoparticles [113].

4. Future Trends:

Futures of using microscopy of nanotechnology in textiles have 2 aspects:
1. Upgrading and promoting available functions,
2. Developing other kinds of microscopy.

Therefore, the progress in microscopic tools can overshadow nano coatings. For example, introducing the forth dimension electron microscopy [114, 115], and scanning transmission electron microscopy [116] can provide the door to observation of different structural, and morphological phenomena of textile surface.

References
Comparative Studies of Polypropylene Nonwoven Sputtered with ITO and AZO.


