

Review

Zinc Oxide for Functional Textile Coatings: Recent Advances

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Abstract: The use of ZnO for the functionalization of textile substrates is growing rapidly, since it can provide unique multifunctional properties, such as photocatalytic self-cleaning, antimicrobial activity, UV protection, flame retardancy, thermal insulation and moisture management, hydrophobicity, and electrical conductivity. This paper aims to review the recent progress in the fabrication of ZnO-functionalized textiles, with an emphasis on understanding the specificity and mechanisms of ZnO action that impart individual properties to the textile fibers. The most common synthesis and application processes of ZnO to textile substrates are summarized. The influence of ZnO concentration, particle size and shape on ZnO functionality is presented. The importance of doping and coupling procedures to enhance ZnO performance is highlighted. The need to use binding and seeding agents to increase the durability of ZnO coatings is expressed. In addition to functional properties, the cytotoxicity of ZnO coatings is also discussed. Future directions in the use of ZnO for textile functionalization are identified as well.

Keywords: ZnO; textile coatings; fiber modification; mechanisms; functional properties

1. Introduction

Functional coatings aim to enhance the properties and performance of textile substrates as well as to introduce new textile functions. To this end, different classical and contemporary organic, organic-inorganic hybrid, and inorganic compounds are used in application processes. Among the latter, ZnO has already been established as a chemical agent for textile functionalization because of its unique physical and chemical properties, environmental friendliness, biocompatibility, and low price. Its important advantage over other materials lies in the fact that bulk ZnO has been generally recognized as a safe (GRAS) substance by the US Food and Drug Administration (FDA) [1].

ZnO is available in the form of a white powder that can be produced from the rare mineral zincite, present in nature, or synthetically from different precursors. In general, ZnO can be applied as a previously prepared ZnO suspension or Zn salt solution, or through the in situ synthesis of ZnO nanoparticles (ZnO NPs) in the presence of a textile substrate. The extraordinary photocatalytic activity, chemical stability under UV radiation exposure, thermal stability, and absorption of a broad range of UV radiation [2–4] allow ZnO particles (ZnO Ps) to be one of the most effective photocatalytic self-cleaning, antimicrobial and UV-protective agents. Furthermore, ZnO Ps have been applied to textile fibers to improve flame retardancy and thermal stability and to achieve moisture management and thermal insulation, electrical conductivity, and hydrophobicity [5–16]. The photocatalytic properties also enable ZnO Ps to be used as degradation agents for different pollutants, such as dyes and surfactants present in textile industry wastewaters [12,17–23].

In the literature, there are several reviews that address ZnO from different perspectives and deliverable highlights. These reviews thoroughly discuss the methods of ZnO synthesis,

precursors used, synthesis conditions, structure characteristics, and properties, as well as the most important application fields of ZnO [2,4,24]. The fundamental mechanism of ZnO photocatalysis, factors affecting the photocatalytic activity, as well as the possibilities and strategies for the improvement of ZnO's photocatalytic performance via metal/non-metal doping, coupling with other semiconductors, heterojunction with nanocarbon components or surface modification were systematically presented [25–28]. In these reviews, the photodegradation efficiency of ZnO in removing contaminants, such as phenolic compounds, persistent organic contaminants, and dyes, was discussed. Furthermore, the biosynthesis of ZnO NPs as an eco-friendly technology without toxic chemicals using different plant extracts, microorganisms and other biomolecules was thoroughly reviewed [29–31]. Since these green routes are especially important for biomedical applications, the photocatalytic, antibacterial, antifungal, drug delivery, anticancer, antidiabetic, anti-inflammatory as well as bioimaging activities of ZnO NPs were taken into consideration [32,33]. However, the antimicrobial activity was most frequently reviewed among the functional properties of ZnO [32–36]. The proposed mechanisms for the antimicrobial action of ZnO NPs against Gram-positive and Gram-negative bacteria were discussed in detail [35].

Review articles dealing with the use of ZnO in textiles are rare [2,37–40]. They are primarily focused on novel application procedures of ZnO [2], being limited to the preparation of antibacterial cellulose/ZnO composites [39] or antimicrobial fabrics containing metal-based nanoparticles, among them ZnO NPs [40]. Existing articles discuss the applications of nanomaterials to textile substrates to obtain different functional properties [38] or give an overview of the development of photocatalyst-modified textiles more generally [37]. To the best of our knowledge, there is no review article that gives comprehensive insights into the functionalities that can be obtained with the application of ZnO to textile substrates. Therefore, the aim of this review is to focus on the recent development of ZnO-functionalized textile fibers, with an emphasis on photocatalytic self-cleaning and purification, antimicrobial and UV-protection activities, flame retardancy, thermal insulation and moisture management, hydrophobicity, and electrical conductivity (Figure 1). The mechanisms of action of ZnO that impart individual properties, as well as various parameters influencing ZnO's performance and efficiency on textile fibers, are also discussed.



Figure 1. Schematic presentation of the functional properties of ZnO on textile fibers.

2. Characteristics of ZnO

ZnO is an n-type semiconductor with a direct and wide band gap in the near-UV spectral region (≥ 3.37 eV) and high electron mobility [41,42]. The ZnO electronic structure provides photocatalytic activity, giving ZnO unique multifunctional properties. When ZnO is excited by a radiation of energy equal to or greater than the band gap (E_g), excited electrons move to the conduction band (e_{CB}^-), leaving behind holes in the valence band (h_{VB}^+). This process is schematically presented in Figure 2.

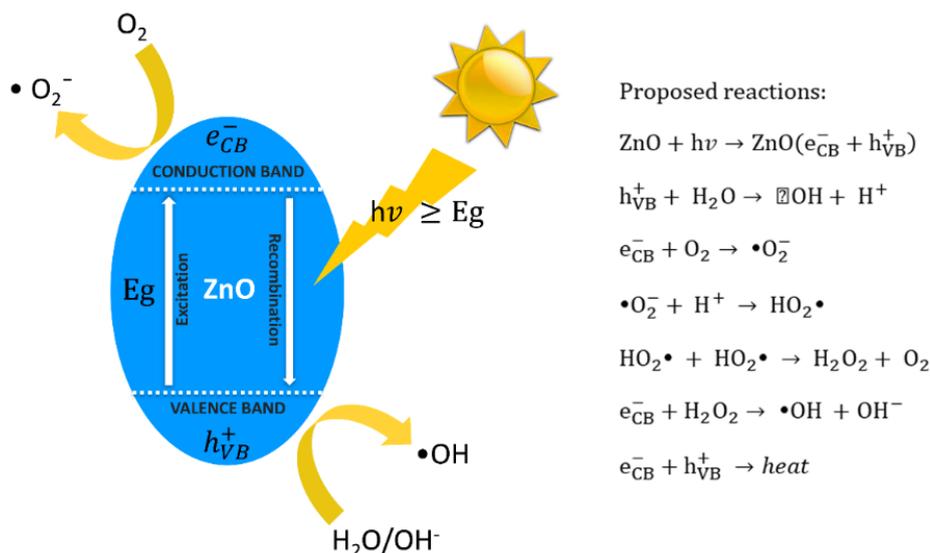


Figure 2. Schematic presentation of ZnO's photocatalytic mechanism.

In the presence of water and oxygen, at least two photochemical reactions occur simultaneously at the ZnO surface. The first is oxidation, in which photo-induced positive holes are involved, and the second is reduction, in which photo-induced negative electrons are involved. In these reactions, highly reactive oxygen species (ROS), i.e., $\bullet\text{OH}$ and $\bullet\text{O}_2^-$, are formed, which are crucial for the photocatalytic efficiency of ZnO. On the other hand, the recombination of the photo-generated holes in the valence band with the photo-excited electrons in the conduction band, which results in the dissipation of heat, which impairs ZnO's photocatalytic effectiveness.

ZnO Ps can be produced by two main approaches, namely, the top-down approach, which is also called the metallurgical process and is based on the roasting of the rare mineral zincite, and the bottom-up approach, which includes various physical and chemical synthesis techniques, among which mechanochemical, electrochemical, sonochemical, hydrothermal, solvothermal, ultrasonic, microwave irradiation, sol-gel, microemulsion, coagulation, controlled precipitation methods, and others, have already been introduced [2,4,28,39,43]. To avoid the use of non-eco-friendly, toxic, and unsafe reagents, the green synthesis of ZnO Ps has become an important alternative to conventional physio-chemical processes. In this eco-friendly approach, the reducing agents, capping agents, dispersants, and binders are replaced with plant extracts and different microorganisms [4,26,29–31,44,45]. To improve the photocatalytic activity and overcome the photo-corrosion of ZnO, structure modification of ZnO Ps has been performed with the aim of minimizing the recombination loss of the photo-induced electron-hole pairs and extending the spectral response of ZnO to the visible spectrum [27]. Structure modification strategies mainly include doping techniques to modify ZnO's properties by incorporating impurities such as metals or non-metals, the coupling of ZnO with other semiconductors, and the coupling of nanocarbon materials to ZnO [25,27,46–50].

The synthesis parameters and conditions, such as synthesis type, precursors used, molar ratio of starting materials, reaction medium, pH value, temperature, type of reducing agent, synthesis time, and drying time, all directly affect the structure, shape, and size of ZnO Ps and consequently their functional properties [2–4,31,45,51]. ZnO generally crystallizes into three main structures, i.e., hexagonal wurtzite, cubic rocksalt, and cubic zinc blende structures, among which the wurtzite crystal structure is the most common since it is thermodynamically stable under ambient conditions [52]. ZnO can be present in a wide diversity of shapes, such as nanocombs, nanorings, nanohelices/nanosprings, nanobelts, nanowires, and nanocages [53]; needle-like, flower-like, rod-like, flake-shaped, and spherical nanodiscs [28]; crushed stone-like, cylinder-shaped, and bullet-like structures; sheets, polyhedra, and ellipsoids [2]; hexagonal plate-like structures [54,55]; rod-shaped particles [54,56]; pyramid-shaped structures [57]; multispheres [58]; doughnut-shaped structures [59–61]; nanosheets [62]; dumbbell-shaped structures [34]; nanoleaves [63]; nanobows [64]; and star, multi-pod and spike-shaped structures [65].

3. Textile Functionalization

To generate ZnO-functionalized textile substrates, different application procedures can be used. Among them, coating processes such as dip-coating, sol-gel, padding, electro deposition, and chemical bath deposition are usually performed. In these application methods, nano- and micro-sized ZnO is mostly deposited on a given textile substrate as a previously prepared suspension, or ZnO is synthesized in situ with the use of solvothermal, hydrothermal, precipitation, ultrasonic, mechanochemical, micro-wave and sol-gel techniques. Table 1 summarizes the most recent coating methods for the fabrication of different functional properties by the modification of textile substrates with ZnO.

Table 1. Summary of the most recent synthesis procedures and coating methods using ZnO to impart functional properties to textile substrates.

Textile Substrate	Synthesis Process	Shape of Particles/Structures	Size ^(a) of Particles/Structures	Application Method	Additional Treatment	Functionality	Ref.	
Cotton	biosynthesis	spherical	53–69 nm	pad-dry-cure	acrylic binder in the functionalization process	antimicrobial activity, photocatalytic degradation	[66]	
	biosynthesis	spherical	10–45 nm	pad-dry-cure	no additional treatment	antibacterial activity, UV protection	[67]	
	biosynthesis	spherical, rod-like	30–80 nm, L: 100 nm, D: 5 nm	dip-coating	no additional treatment	antibacterial activity	[68]	
	biosynthesis	hexagonal, rod-like	10–42 nm, 8–38 nm	pad-dry-cure	no additional treatment	UV protection, antimicrobial activity	[69]	
	green synthesis	spherical, irregular	39.34 nm, 43.63 nm	pad-dry-cure	chitosan in the functionalization process	hydrophobicity, UV resistance, antibacterial activity	[11]	
	precipitation	rods, sheets, flake-like, flower-like	not specified	dip-coating + microwave	no additional treatment	UV protection, photocatalytic self-cleaning	[13]	
	precipitation	star-like	0.5–1 μ m	dip-coating	no additional treatment	UV protection	[70]	
	precipitation	not specified	not specified	interfacial polymerization into microcapsules, pad-dry application	tetraethoxysilane and hexadecyltrimethoxysilane after-treatment	UV protection, thermal insulation, superhydrophobicity	[71]	
	co-precipitation	hexagonal	47.2 nm	pad-dry-cure	poly-hydroxy-amino methyl silicone binder in the functionalization process	UV protection, antimicrobial activity	[72]	
	chemical precipitation	hexagonal	24–28 nm	pad-dry-cure	NaOH after-treatment	UV protection, antimicrobial activity	[73]	
		commercially available ZnO (not specified)			dip-pad-dry-cure	no additional treatment	photocatalytic degradation (Self-cleaning)	[74]
		commercially available ZnO (not specified)			sonochemical coating	crosslinking agent (galic acid) in the functionalization process	antimicrobial activity	[75]
		commercially available ZnO (not specified)			pad-dry	inductively coupled (RF) plasma pretreatment	antibacterial activity	[76]
		commercially available ZnO (not specified)			pad-dry-cure	inductively coupled (RF) plasma pretreatment	UV protection	[77]
		commercially available ZnO (not specified)			dip-coating	1-butyl 3-methyl imidazolium chloride in the functionalization process	antibacterial activity, UV protection	[78]

Table 1. Cont.

Textile Substrate	Synthesis Process	Shape of Particles/Structures	Size ^(a) of Particles/Structures	Application Method	Additional Treatment	Functionality	Ref.
Cotton			commercially available ZnO (not specified)	dip-coating	octadecanethiol in the functionalization process or as an after-treatment	superhydrophobic, antibacterial activity	[79]
			commercially available ZnO (not specified)	pad-dry-cure	organic/inorganic binder, repellent chemical in the functionalization process	UV protection, superhydrophobic, antibacterial activity	[80]
			commercially available ZnO (not specified)	dip-coating	myristic acid in the functionalization process	electrical conductivity	[81]
	hydrothermal	Hexagonal	56 nm	dip-coating	fluoro-surfactant as stabilizer in the synthesis process	photocatalytic degradation	[12]
	in situ	star-like	55–70 nm	dip-coating + sonication	Tragacanth gum in the functionalization process	photocatalytic degradation	[42]
	in situ	layers, spherical	not specified	in situ dip-coating	no additional treatment	UV protection	[51]
	in situ	spherical, elliptical	37 nm	in situ sol-gel	no additional treatment	antibacterial activity	[82]
	in situ	not specified	198.5 nm, 359 nm, 2520 nm	in situ dip-coating	hexamethyltriethylenetetramine in the functionalization process	antibacterial activity, UV protection	[83]
	in situ	Hexagonal	35 nm	pad-dry-cure	no additional treatment	antibacterial activity, UV protection	[84]
	in situ	rod-like, spherical, plate-like	not specified	in situ sol-gel	no additional treatment	antibacterial activity	[85]
	in situ	Spherical	97 nm	pad-dry-cure	NaOH pretreatment	antibacterial activity, UV protection	[86]
	in situ	nanoparticles, nanorods	48–62 nm	in situ dip-coating	Cu ₂ O, folic acid	UV protection	[87]
	solvothermal	rod-like	1.76 ± 0.12 μm	dip-coating	polydopamine template as a pretreatment, hexamethylenetetraamine in the functionalization process	antimicrobial activity, UV protection	[88]
	solvothermal	rod-like	not specified	dip-coating	hexamethylenetetraamine in the functionalization process	UV protection, electrical conductivity	[89]
	sol-gel	hexagonal	18 nm, 19 nm	sputter seed layer deposition and sol-gel	NaOH pretreatment, hexamethylenetetraamine in the functionalization process	UV protection, electrical conductivity	[90]
	ultrasonic	flake-like, flower-like, sheets, hexagonal	not specified	dip-coating + ultrasonic irradiation	sodium dodecyl sulfate as a pretreatment, different surfactants in the functionalization process	antimicrobial activity	[91]
	ultrasonic	needle-like, bramble-like	not specified	ultrasonic irradiation	sodium dodecyl sulfate as a pretreatment	electrical conductivity	[10]

Table 1. Cont.

Textile Substrate	Synthesis Process	Shape of Particles/Structures	Size ^(a) of Particles/Structures	Application Method	Additional Treatment	Functionality	Ref.
Cotton	not specified	not specified	50 nm	pad-dry-cure	acrylic binder in the functionalization process	UV protection	[92]
	not specified	not specified	not specified	atomic layer deposition/molecular layer deposition	pretreatment with Al ₂ O ₃ seed layer deposition, hydroquinone in the synthesis process	electrical conductivity	[93]
Cotton/ polyester	in situ sonosynthesis	semi-hexagonal nanosheets, cobblestone-like nanoparticles	56 nm, 20 nm	in situ dip-coating + ultrasonic irradiation	Fe ₃ O ₄ , cetyltrimethylammonium bromide dispersing agent in the synthesis process	self-cleaning, antimicrobial activity	[94]
Flax	in situ	spherical, platelets	58.3–223.9 nm, 600–684.2 nm	in situ dip-coating	deposition of Ag NPs as pretreatment	antibacterial activity, hydrophobicity, UV resistance	[95]
Jute	co-precipitation	not specified	38–60 nm, 30–500 nm	pad-dry	potassium methyl silicate in the functionalization process	fire retardancy	[5]
	co-precipitation	rod-like	38–60 nm	pad-dry-cure	hydroxymethyl amino silicate binder in the functionalization process	fire retardancy	[96]
Jute	solvothermal	rod-like	L: 2.5 µm, D: 140 ± 44 nm	dip-coating (seeding, growth)	hexamethylenetetramine in the functionalization process	hydrophobicity	[97]
Polyamide	hydrothermal in the presence of hexamethylenetetramine	rod-like	not specified	dip-coating (seeding, growth)	screen printing of Ag as a pretreatment	electrical conductivity	[98]
Polyamide, polyester, polypropylene	not specified	microrods	L: 3.9 ± 0.4 µm, D: 252 ± 5 nm; L: 5.6 ± 0.2 µm, D: 389 ± 8 nm; L: 4.1 ± 0.6 µm, D: 313 ± 2 nm	hydrothermal deposition	hexamethylenetetramine in the functionalization process	antibacterial activity	[99]
Polyester	chemical bath deposition	rod-like	L: 1 µm, D: 450 nm	chemical bath deposition	hexamethylenetetramine in the functionalization process	antimicrobial activity	[100]
		commercially available ZnO (not specified)		dip-coating	NaOH pretreatment or in the functionalization process	self-cleaning, antibacterial activity	[101]
		99.99% zinc target		RF magnetron sputtering	deposition of Ag film using DC magnetron sputtering before ZnO	photocatalytic degradation	[102]
	hydrothermal	rod-like	L: 1 µm, D: 50 nm	seed layer deposition	plasma pretreatment, hexamethylenetetramine in the functionalization process	photocatalytic degradation, self-cleaning	[103]
	Mechanochemical	not specified	30–60 nm	dip-coating + shaking	NaOH and UVC irradiation pretreatment	photocatalytic degradation	[104]
	sol-gel	irregular	~40 nm	dip-coating + shaking	no additional treatment	photocatalytic degradation	[105]

Table 1. Cont.

Textile Substrate	Synthesis Process	Shape of Particles/Structures	Size ^(a) of Particles/Structures	Application Method	Additional Treatment	Functionality	Ref.
Polyester	sol-gel in situ mineralization	wurzite	not specified	sol-gel in situ mineralization	radiation induced graft polymerization of c-methacryloxypropyl trimethoxysilane in the pretreatment process	UV resistance, hydrophobicity, thermal resistance	[106]
	Solvothermal	spherical	34.12 nm	dip-coating	cetyltrimethylammonium bromide, urea and corn silk in the functionalization process	antimicrobial activity	[16]
	Ultrasonic	spherical	52.6 nm	dip-coating + ultrasonic irradiation	no additional treatment	photocatalytic self-cleaning, antibacterial activity	[107]
	not specified	not specified	not specified	atomic layer deposition	cupric nitrate trihydrate, dimethylformamide, 1,3,5-benzene-tricarboxylic acid, anhydrous ethanol in the synthesis process	photocatalytic degradation	[108]
Polyester/cotton	sol-gel	not specified	50, 130, 260, 380, 650 nm	pad-dry-cure	no additional treatment	antimicrobial activity, moisture management	[14]
Polypropylene, cotton	commercially available ZnO (not specified)			pulsed laser deposition	RF plasma pretreatment	antimicrobial activity	[15]
Polyurethane	Hydrothermal	nanoneedles	<i>L</i> : 2–3 μm , <i>D</i> : 30 nm	dip-coating	polydopamine pretreatment	photocatalytic degradation, antimicrobial activity	[109]
Silk	in situ	rod-like	<i>L</i> : 0.9–13 μm , <i>D</i> : 70–160 nm	in situ pad-dry	after-treatment with n-octadecenethiol	UV protection, superhydrophobicity	[110]
	in situ	nanorod arrays	<i>D</i> : 100–200 nm	electro-deposition	no additional treatment	electrical conductivity	[111]
	in situ	tower-like nanowires	not specified	cathodic deposition	Au-metallization as a pretreatment	electrical conductivity	[9]
Sisal	precipitation	not specified	not specified	dip-coating	butane tetracarboxylic acid in the functionalization process	fire retardancy	[6]

^(a) *L*—length, *D*—diameter.

3.1. Photocatalytic Self-Cleaning Properties

The self-cleaning properties of ZnO Ps are derived from their photocatalytic performance [13,25,28,37,112–115]. If ZnO Ps are present on the surface of textile fibers, ZnO is capable of photodegrading various organic colored and colorless dirt stains when it encounters them [116]. In this case, the direct photooxidation of the organic compounds by reaction with h_{VB}^+ and the indirect photooxidation of the organic compounds by reaction with ROS occur [75,117]. This leads to the decomposition of organic compounds and their removal from textile fibers. Since the textile surface is cleaned without the use of washing processes, this phenomenon is called photocatalytic self-cleaning.

The self-cleaning activity of ZnO Ps has mostly been studied on ZnO surface-modified cotton [13,107,114,118] and polyester [101,105,115] fibers, as well as cotton/polyester blends [94]. As stains, coffee, tea, and the dyes methylene blue and rhodamine B were applied to the textile fabric surface before exposure to solar and UV light irradiation for different intervals under different humidity conditions. The self-cleaning efficiency of ZnO was determined based on the color difference measurement between the irradiated and non-irradiated stained samples or by the observation and calculation of color intensity. To determine the durability of the photocatalytic self-cleaning properties of the coating, dyes or colored stains were consecutively applied to the same position on the sample surface.

The results showed that the self-cleaning properties depended on the content of ZnO Ps. A higher concentration of ZnO Ps resulted in increased photocatalytic degradation [94,105,115,118]. A treatment of polyester with sodium hydroxide to increase the surface area and wettability of fibers enhanced the adsorption of ZnO Ps and consequently the self-cleaning degradation of methylene blue [101,107]. The self-cleaning effect was also enhanced with increasing relative humidity, indicating that the water molecules present at the surface of the ZnO Ps contribute to ROS formation [13]. Accordingly, the highest self-cleaning effect was achieved at 90% relative humidity, where coffee stains were almost completely removed from cotton fabric after 15 h of exposure without washing.

Furthermore, the photocatalytic activity of ZnO was enhanced when coupled with other semiconductors or carbon materials [94,118]. Accordingly, if $\text{Fe}_3\text{O}_4/\text{ZnO}$ nanocomposites were deposited on a cotton/polyester fabric, an increase in the self-cleaning properties of the sample was obtained with an increase in the concentration of iron sulphate [94]. It was believed that the presence of Fe_3O_4 increased the possibility of the separation of photo-generated charges, prolonging the electron-hole recombination. If a reduced-graphene graphene oxide-ZnO nanocomposite was applied to cotton fabric, excellent photodegradation of methylene blue dye and tea stains was observed, even under sunlight irradiation [118].

ZnO-coated textile substrates have previously been used for the solar-driven photodegradation process of toxic organic compounds in water and air. To this end, ZnO-coated polyester [21,103,104,119] or cotton [12] substrates were immersed in different dye solutions, such as methylene blue, methyl orange, C.I. Acid Blue 9, C.I. Reactive Orange 13 and C.I. Reactive Violet 5, and the photocatalytic degradation of the dye was monitored spectrophotometrically during UV and solar light irradiation. The decomposition of the dye chromogen resulted in solution decolorization, reflecting a decrease in the dye absorbance (Figure 3).

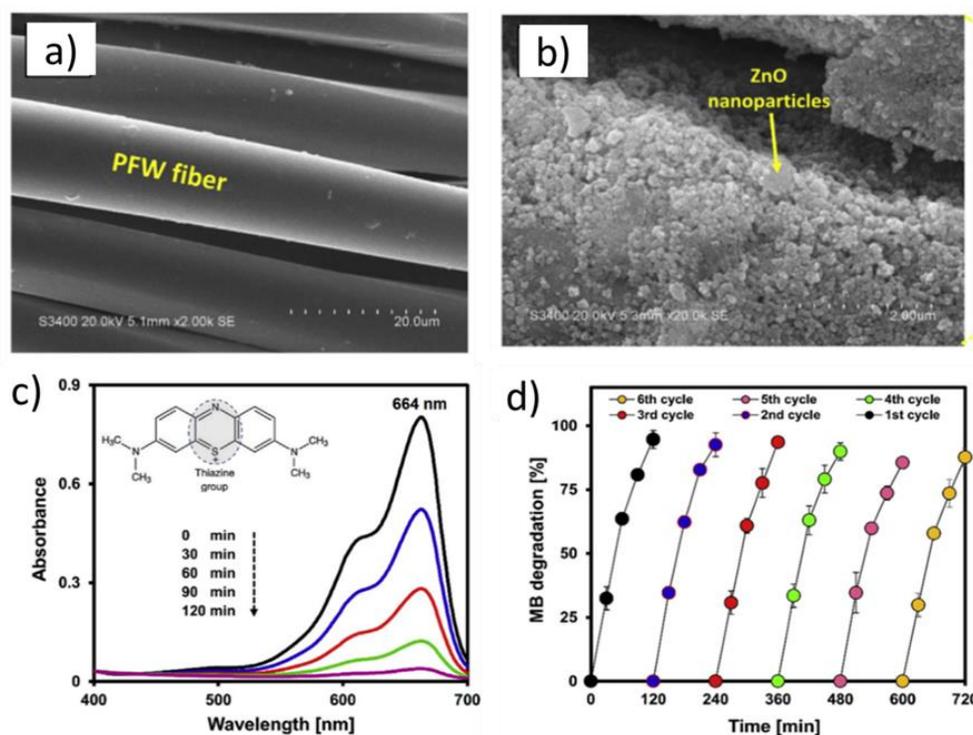


Figure 3. Untreated (a) and ZnO-coated (b) polyester fibers; UV-Vis absorption spectra of methylene blue solution in the course of photocatalysis by ZnO-coated polyester fabric (c); reusability test of ZnO-coated polyester fabric for methylene blue degradation (d). Reprinted with permission from [105]. Copyrights 2017 Elsevier.

The results also showed that the photocatalytic degradation activity of ZnO-coated textile substrates increased with increasing surface porosity, roughness, specific surface area, and hydrophilicity [12,21]. These factors facilitate the adsorption of the dye onto the ZnO Ps and enhance the formation of ROS. On the other hand, an increase in the initial concentration of dye decreased the rate of ZnO photocatalytic degradation [119]. The reason for this was attributed to the blocking of the active sites by the dye molecules, which also prevent UV rays from reaching ZnO Ps. The photocatalytic performance of ZnO Ps slightly decreased after successive dye degradation cycles due to the liberation of ZnO Ps from the surface of the textile substrate [105].

Beside dyes, the effective photocatalytic degradation of other toxic compounds such as p-nitrophenol [120], organophosphate methyl parathion [34], and formaldehyde [102], in the presence of ZnO- or ZnO nanocomposite-modified fabrics, was observed. The application of silver/ZnO composite films onto polyester fabric improved the photocatalytic activity of ZnO, resulting in increased formaldehyde degradation rates compared to fabrics coated only with ZnO film [102].

It was found that the mechanism of pollutant degradation by ZnO Ps depends directly on the pollutant chemical structure. Accordingly, when studying the mechanism of methylene blue and formaldehyde degradation in the presence of different chemical scavengers, it was found that $\bullet\text{OH}$ radicals play a vital role in the dye oxidation reaction [105] and that $\bullet\text{O}_2^-$ plays a vital role in formaldehyde degradation [120]. On the other hand, hydrolysis was assumed to be the major mechanism for the photocatalytic degradation of the organophosphate methyl parathion in the presence of ZnO [34].

3.2. Antimicrobial Properties

The antimicrobial activity of ZnO is attributed to ROS generated through ZnO photocatalysis and to Zn^{2+} cations liberated from the surface of ZnO Ps. Despite much research in this field, the

exact mechanism of the antimicrobial activity of ZnO is still not completely understood [36,100]. The reason for some contradictory results most likely arises from the use of different zinc precursors, synthesis conditions, ZnO concentrations, ZnO crystal structures, sizes, shapes, surface textures, defects, and functionalizations, which influence the antimicrobial mechanism [36]. The different proposed mechanisms of antimicrobial activity of ZnO Ps are presented in Figure 4. Because of these multiple simultaneous antimicrobial mechanisms of action, it is much more difficult to develop microbial resistance against ZnO.

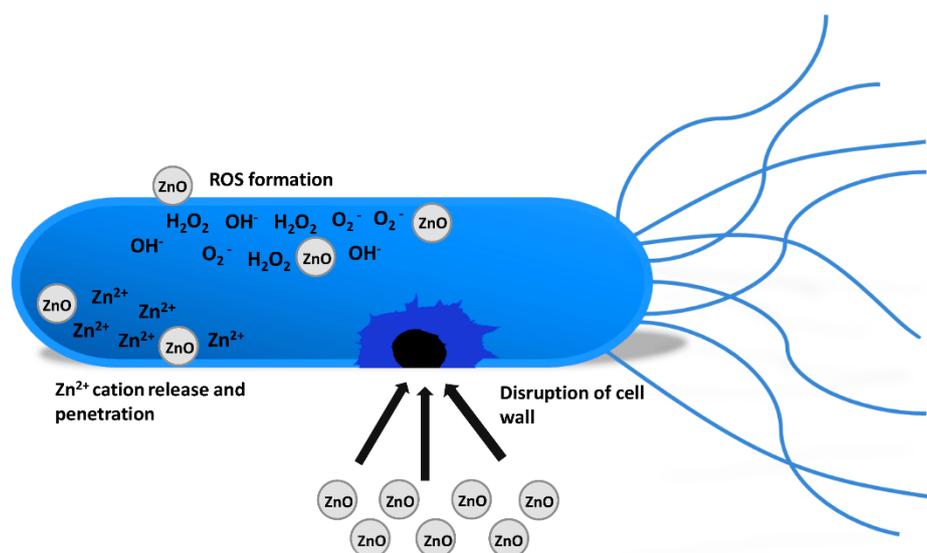


Figure 4. Schematic presentation of different mechanisms of antimicrobial activity of ZnO Ps.

It is assumed that the photocatalytic generation of ROS, especially H_2O_2 and $\bullet\text{OH}$, is crucial for the antimicrobial activity of ZnO under UV and visible light exposure [26,32,35,36,100,121]. When ZnO NPs encounter bacterial cells, they can directly adsorb to the cell surface and disrupt the cell wall. The generated highly reactive ROS can easily incorporate into the bacterial cell membrane, causing the disruption of cellular components, such as DNA, lipids, and proteins, via oxidative stress. Furthermore, Zn^{2+} cations released from ZnO dissolution can also penetrate the bacterial cell, where they inhibit the action of respiratory enzymes. Under dark conditions, the antimicrobial activity of ZnO is assumed to be less related to the generation of ROS and is mainly attributed to the attachment of ZnO NPs to bacterial cell walls and increasing concentrations of Zn^{2+} cations in the bacterial cytoplasm [121].

To create antimicrobial textiles, cellulose fibers represent the most attractive textile substrate for the application of ZnO Ps [11,32,42,66,68,69,72,73,76,82,84–86,88,90,122–127]. In addition to cellulose, ZnO Ps have been applied to different synthetic fibers, such as polyester [16,99,100,128], polypropylene [15,99,129], polyamide [99], polyurethane [109] and cellulose/polyester blends [14,94].

The antibacterial activity of textile fibers has been most often tested against *Staphylococcus aureus* Gram-positive bacteria [14–16,42,67,68,72,75,76,82,94,99,126,127,129] and *Escherichia coli* Gram-negative bacteria [15,16,42,67,68,76,94,99,109,124,126,127,129]. In addition, testing against methicillin-resistant *Staphylococcus aureus* (MRSA) [82], *Staphylococcus epidermidis* [82], *Propionibacterium acnes* [82], *Candida albicans* [16,42,94,124], *Bacillus subtilis* [67], *Pseudomonas aeruginosa* [67], *Saccharomyces cerevisiae* [125], *Gluconobacter cerinus* [88], and *Klebsiella pneumonia* [72] has been performed. The antifungal activity has been tested against *Aspergillus flavus* [126].

The results of the antibacterial efficiency of ZnO Ps showed that at lower concentrations, ZnO Ps were more effective against Gram-positive than Gram-negative bacteria (Figure 5) [11,15,67,85,100], since the former are more susceptible to inhibition by ZnO Ps than the latter. However, the growth inhibition of Gram-negative bacteria significantly increased at higher ZnO concentrations and with longer contact times, resulting in excellent antimicrobial activity against both Gram-positive and

Gram-negative bacteria [84,99]. A difference in the susceptibility of Gram-positive and Gram-negative bacteria to ZnO Ps was attributed to the difference in their bacterial wall structure [25,82]. It is believed that the more complex, multilayered structure of Gram-negative bacteria, with an outer plasma membrane, could provide greater protection against ROS in comparison to the less complex structure of Gram-positive bacteria. However, the reverse results were also presented, where a greater sensitivity of Gram-negative bacteria was attributed to their thinner cell wall peptidoglycan, which could be more easily damaged by ROS than the thicker cell wall of Gram-positive bacteria [68,99].

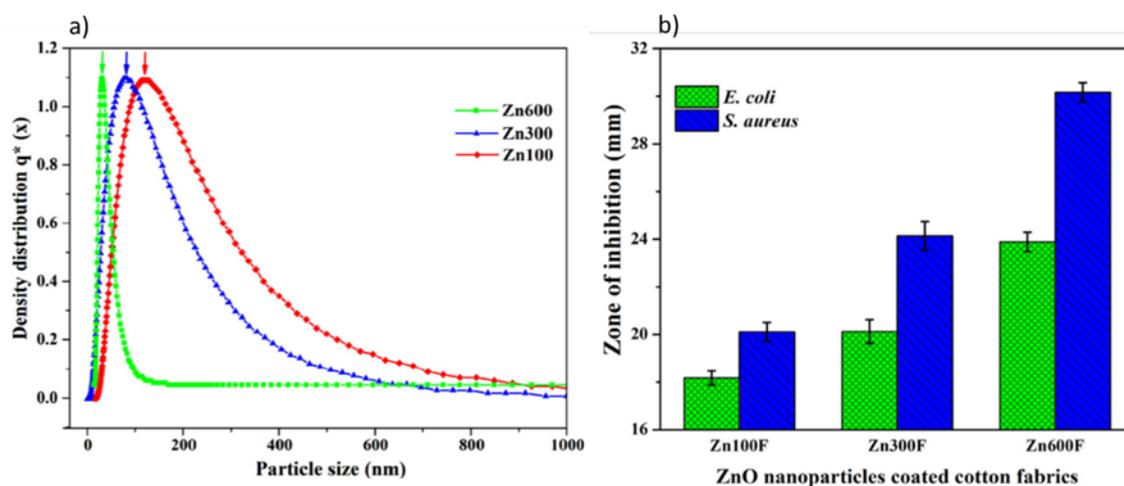


Figure 5. Particle size distribution curves of ZnO NPs (a), antimicrobial activity of ZnO NPs coated cotton fabric (b). Sample codes: Zn600, Zn300 and Zn100 are ZnO nanopowders calcined at 100, 300 and 600 °C, respectively; Zn100F, Zn300F and Zn600F are fabric samples coated with different ZnO nanopowders. Reprinted with permission from [11]. Copyrights 2017 Elsevier.

The antibacterial activity of ZnO Ps on textile fibers is directly influenced by the ZnO concentration [78], particle size [11], crystalline structure [69] and surface-to-volume ratio [99,100], and by the pH of the solution [128] as well as the chemical and morphological properties of textile fibers [99,128]. Increasing ZnO concentration resulted in higher antimicrobial activity [72,85]. Furthermore, a decrease in the ZnO particle size, which consequently increases the particle surface-specific area, significantly enhanced the antimicrobial activity (Figure 5) [11]. It was also observed that an increase in fiber hydrophilicity and surface roughness enhanced the amount of absorbed ZnO Ps, reflecting higher antimicrobial activity [99]. Accordingly, plasma and alkali pre-treatment of textile fibers has already been established in ZnO modification processes to increase both wettability and roughness [77,129,130]. It was also observed that the antimicrobial activity of ZnO NPs is shape-dependent [69]. Nano-rod NPs showed enhanced antibacterial properties against Gram-negative and Gram-positive bacteria compared to hexagonal ZnO NPs.

For textile application, the washing fastness of the antimicrobial coating is of high importance. This property is directly related to the shape and size of ZnO particles and their stabilization and fixation to the fiber surface. To this end, cotton fibers were pre-activated with different fixing agent networks, such as polydopamine [88] or enzymatically crosslinked gallic acid [75], in which ZnO NPs were embedded. A newer amino-silicone binder was also applied in the presence of Zn NPs in a one-step application process onto cotton fibers [72]. The entrapment of ZnO NPs into cotton fabric was increased by the usage of ionic liquid, influencing the enhanced swelling property of the fibers [78]. Different surfactants were used to control the shape and size of ZnO NPs as encapsulated species, and sodium dodecyl sulphate was found to be a very effective stabilizing agent that improved the durability and decreased the leaching of the coated ZnO NPs from cotton fibers [91]. The coating durability was also increased by the in situ synthesis of ZnO NPs within the cotton fibers in the presence of appropriate reducing and stabilizing agents, such as plant extracts, hexamethyltriethylene

tetramine, polymethylol compounds, and functionalized polyethyleneimine, without the support of other capping and binding agents [27,83,86]. In addition to washing fastness, the stability and durability of ZnO particles incorporated into the styrene-acrylic layer on cotton fibers were also measured during long-term storage in wet and hot environments [122]. The results revealed effective antibacterial activity of ZnO particles, even after accelerated ageing.

It was also found that the introduction of ZnO into an organic-inorganic composite composed of epoxy resin and modified Ag/ZnO NPs significantly enhanced antibacterial efficiency as well as provided a sufficient ZnO antifungal activity (Figure 6) [126]. Simultaneously, the covalent bonding of the nanocomposite to the cotton fabric was carried out via interactions between the epoxy groups in the polymer chains and hydroxyl groups on the cotton fiber surface, enhancing its durability.

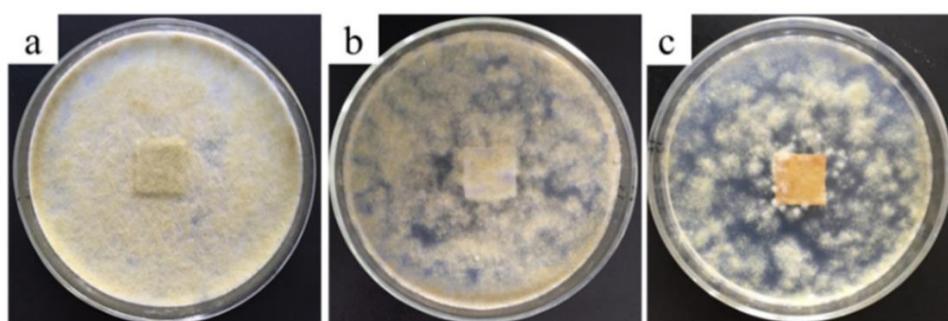


Figure 6. Antifungal activity of native cotton fabric (a), fabric coated with ZnO (b), fabric coated with Ag/ZnO (c). Reprinted with permission from [126]. Copyrights 2019 Elsevier.

In addition to antimicrobial properties, the cytotoxicity of ZnO NPs and ZnO-textile coatings against human and animal cells was evaluated, since these parameters are essential for biomedical applications. It was shown that the cytotoxicity of ZnO NPs depends on both size and concentration. A comprehensive investigation of cytotoxicity and the mechanism of ZnO NPs with three different sizes on a human model cell revealed that the cytotoxicity of ZnO NPs was attributed to the release of Zn^{2+} , induction of oxidative stress and inflammatory response, and that the death mode of the human hepatocyte cells incubated with ZnO NPs was necrotic rather than programmed cell death [131]. It was also found out that the solubility of ZnO NPs is the most important factor in causing cytotoxicity in vitro [132]. While Zn^{2+} at low concentrations is essential for maintaining the cellular processes and metabolism, Zn^{2+} at higher concentrations can cause toxicity. Accordingly, the presence of ZnO NPs at low concentrations in a textile coating did not compromise the cell viability, suggesting their high biocompatibility [75], and the capping of ZnO NPs with plant extracts further reduced their cytotoxicity [86]. A comparison of the effects of different coating procedures on ZnO cytotoxicity showed that the in situ synthesis of ZnO could importantly decrease its cytotoxicity compared to the deposition of previously synthesized ZnO to a polymer matrix [133]. The reason for this was attributed to the good distribution and superior performance of the in situ synthesized ZnO within the polymer matrix in comparison with the previously synthesized and agglomerated ZnO, causing its decreased cytotoxicity. On the other hand, a recent study on bird representatives confirmed the hypothesis that even at small doses (i.e., environmentally relevant doses) and for short exposure periods, ZnO NPs induce erythrocyte changes suggestive of mutagenic and cytotoxic effects, requiring additional studies to help better understand the health hazards and risks of ZnO NPs [134].

3.3. UV protection

The UV protection properties of ZnO are derived from its excellent chemical stability under UV radiation exposure and high effectiveness in blocking both UV-A and UV-B radiation [3]. The proposed mechanism of UV protection in ZnO involves the absorption of UV radiation as well as the refraction and/or scattering of the UV rays through its high refractive index, which prevents direct and

diffuse transmittance of UV rays through textile material to reach the skin (Figure 7). The efficiency of UV protection is expressed in terms of the ultraviolet protection factor (UPF) [135], where according to the AS/NZ 4399 [136] and EN 13758 standards [137], excellent protective properties are obtained with UPF values of 40–50 or higher and with UVA average transmission smaller than 5%.

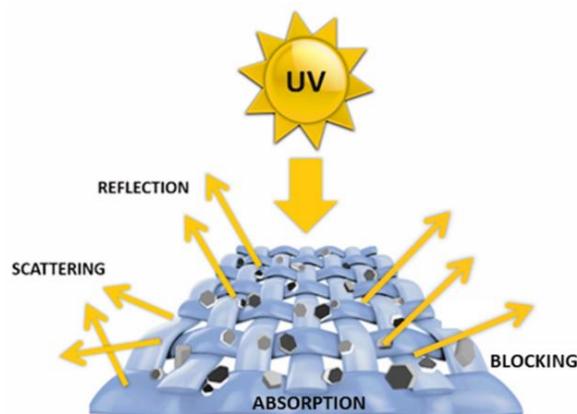


Figure 7. Schematic presentation of the UV protection mechanism of ZnO particles on textile fibers.

For human skin protection against UV irradiation, ZnO has mostly been applied to woven and knitted cotton fabrics, since natural cellulose fibers are usually used for the tailoring of light summer protective clothing [11,71–73,77,78,80,83,84,86–88,90–92]. Research on other textile substrates has been presented as well [106,110,129].

The efficiency of the UV protection of ZnO is strongly dependent on its concentration, particle size and shape. As expected, an increased concentration of ZnO in the textile substrate enhances the UV protection properties [51,71,72,78,84]. Furthermore, ZnO NPs maintained much higher UV protection than micro-sized ZnO Ps because of their high specific surface area and refractive index [11,129]. Furthermore, the application of previously prepared ZnO suspensions, in which particles were agglomerated, could not preserve the excellent UV protection properties since the agglomeration phenomenon significantly hindered the UV-blocking efficiency [73,92]. To decrease the particle size and avoid particle agglomeration, in situ synthesis of ZnO NPs in the presence of appropriate reducing agents as well as stabilizing and capping agents has been recommended to achieve excellent UV protection [51,83,88]. Among stabilizing and capping agents, different chemicals, such as hexamethyltriethylene tetramine [83], polydopamine (Figure 8) [88], folic acid [87], plant extracts [11,73], proteins isolated from fungus [67], and surfactants [83] have been used in the ex situ and in situ syntheses of ZnO NPs. Some stabilizing agents, such as plant extracts, also exhibit UV-absorbing and UV-blocking properties themselves [73,138]. Another important advantage of the in situ synthesis of ZnO NPs is the good adhesion between NPs and textile substrates, which consequently improves the durability of UV protection.

To enhance the absorptivity of ZnO, textile fibers were pre-treated with plasma [77]. An increase in the ZnO-textile substrate adhesion and a prolonged coating durability were provided by the usage of binding [72] and capping [86] agents. When methacryloxypropyl trimethoxysilane was used as a binding agent, a covalent bonding of ZnO to the organic-inorganic hybrid polymer film exhibited excellent washing fastness in the coating, even after 40 washing cycles [106].

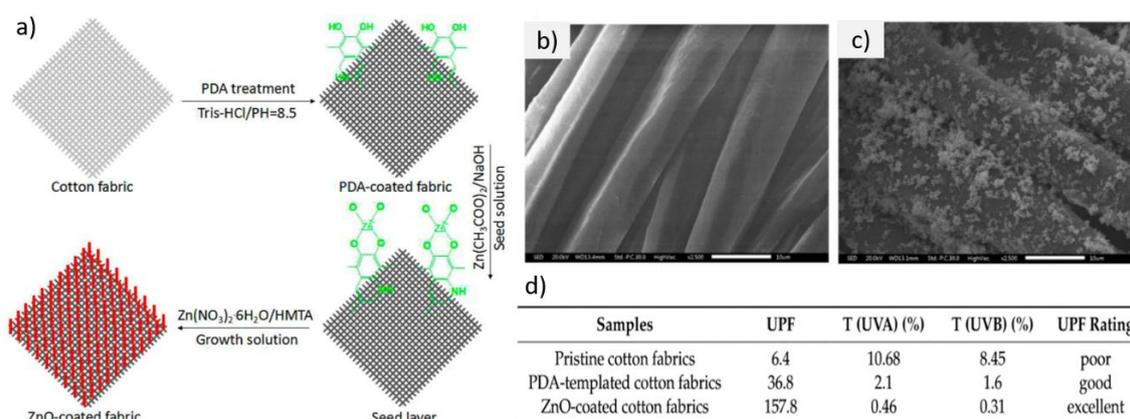


Figure 8. Schematics of the growth of ZnO NPs on PDA-templated cotton fabric (a); surface morphology of pristine (b) and ZnO-coated (c) cotton fibres; UPF values, transmission (T) in the UVA and UVB regions, and UPF rating of the cotton fabric samples (d).

3.4. Thermal Insulation and Moisture Management

The presence of ZnO on fibers can contribute considerably to the wear comfort performance of textiles, since it can significantly increase the thermal insulation and improve the moisture management capability of fabrics. Accordingly, a cotton fabric with increased thermal insulation for cold weather clothing was created by covering the pores of twill woven fabric using a ZnO coating [139]. Consequently, the water vapor permeability of the fabric decreased, and the resistance to evaporative heat loss increased significantly. Furthermore, the thermal insulation properties of cotton fabric were increased by coating the fibers with aluminum-doped ZnO-embedded lemon microcapsules due to the decreased transmittance in the visible and near-infrared region. This performance was gradually enhanced with increased microcapsule loading [71].

Moisture management properties were developed on polyester/cotton woven fabric for sportswear applications by treating it with ZnO NPs of different particle sizes [14]. It was observed that the moisture flow speed increased with decreasing ZnO particle size. It was believed that smaller particles created finer capillaries in the fabric structure, allowing for faster moisture transport than in the case of larger capillaries present between larger particles. Furthermore, a fabric with superior moisture management can successfully remove sweat, thus preventing bacterial growth on the human body [14]. To create moisture-wicking dual-layer hydrophilic-hydrophobic textiles, ZnO NPs were covalently attached onto an electrospun hydrophobic poly(vinylidene fluoride) inner layer to induce a push-pull effect to improve water transport behavior [140]. At the same time, the antimicrobial behavior of ZnO was also beneficial.

3.5. Flame Retardancy

It has been proposed that the flame retardancy of ZnO is based on a condensed phase mechanism of action via the heat barrier effect [141,142]. As a highly thermally stable inorganic material, ZnO can protect the insulation layer on the fiber surface, which reduces the transfer of heat, fuel, and oxygen between the flame and fibers and consequently reduces the rate and intensity of combustion. ZnO has already been designated as a smoke suppressant [142].

There are only a few papers involving ZnO-based flame-retardant textiles. In these studies, ZnO alone or in combination with organic phosphorus compounds was applied to cotton, polyester or cotton/polyester blends [7,84,142–144], but for technical applications, jute and sisal fibers were also used as textile substrates [6,96]. The results showed that the flame retardancy of ZnO-coated cellulose fibers was significantly enhanced if bulk ZnO was replaced with ZnO NPs [6]. The presence of ZnO increased the limiting oxygen index of the cellulose fibers [6,96], reduced the heat release rate, and acted as a smoke suppressant [144]. Whereas the thermal stability of cellulose fibers was not improved

by ZnO, the amount of char residue was significantly increased [13,96,142]. In contrast, the thermal stability of ZnO-treated polyester fibers even decreased compared to untreated fibers [143]. However, the loading of ZnO onto an organic-inorganic hybrid soloxane polymer film significantly modified a decomposition pathway of the coating, which significantly enhanced the thermal stability of the polyester fibers [106]. Furthermore, even at high mass loadings, ZnO coating could not preserve the self-extinguishing behavior of cellulose fibers [84,142], but the after-flame time was shorter, and more char was formed in comparison with untreated fibers (Figure 9) [84].

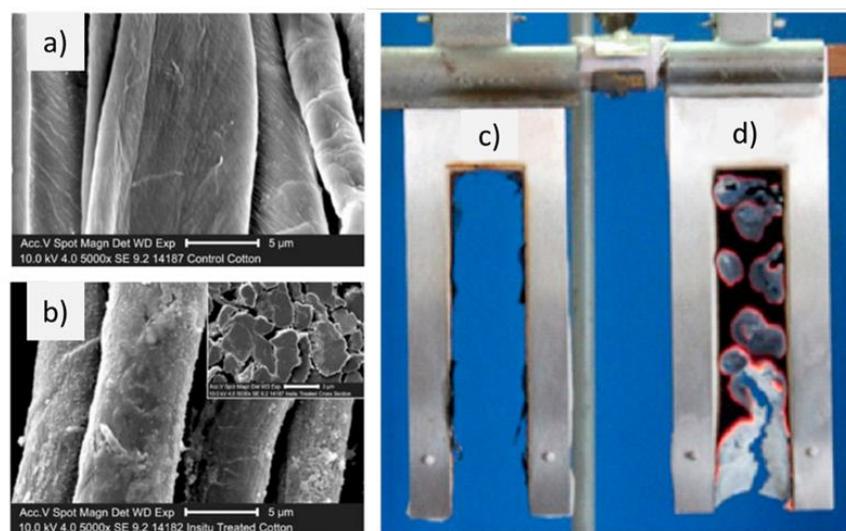


Figure 9. Surface morphology of control (a) and ZnO-treated (b) cotton fibres; flammability test of control (c) and ZnO-treated (d) cotton fabric. Reprinted with permission from [84]. Copyrights 2017 Springer.

3.6. Hydrophobicity

For textile applications, the hydrophobic behavior of ZnO Ps is less relevant, since the hydrophilic self-cleaning activity of ZnO with a high water absorption capacity is increasingly exploited. It was found that the synthesis conditions directly influenced the morphology of ZnO crystals and consequently the hydrophobicity/hydrophilicity of cotton fabric [13]. Namely, ZnO crystals with rod structures that were grown in a seeding solution at pH 4–5 were more hydrophobic compared with the flake- and flower-like ZnO structures which preferentially formed at pH 6–7 and pH 10–11 (Figure 10).

According to the literature, several studies have been performed in which ZnO Ps were used in combination with hydrophobic or oleophobic precursors to provide multifunctional antimicrobial, UV-protective, and superhydrophobic properties on the textile surface [12,71,79,80,97,106,110,145]. In these studies, ZnO NPs contributed to the development of superhydrophobic properties by providing hierarchical micro- and nano-scale rough structures at the fabric surface (Figure 11), representing a crucial morphological factor for the creation of hydrophobic self-cleaning effects called the “lotus effect”. Regarding further morphological changes, the chemical modification of fibers to ensure low-surface-energy hydrophobicity, without which the “lotus effect” would not be achieved, was performed by the application of organic or inorganic-organic hybrid precursors with alkyl [71,79,97,106,110,145] or perfluoroalkyl [12,80] functional groups.

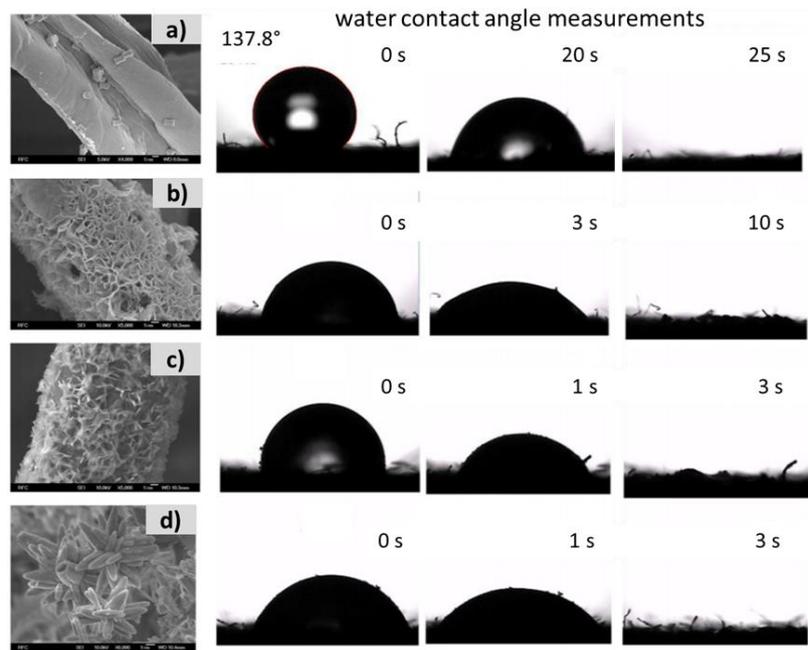


Figure 10. Surface morphology of ZnO-coated cotton fabric synthesized at pH 4–5 (a), pH 6–7 (b), pH 8–9 (c), pH 10–11 (d) and the time dependence of water contact angles. Reprinted with permission from [13]. Copyrights 2017 Elsevier.

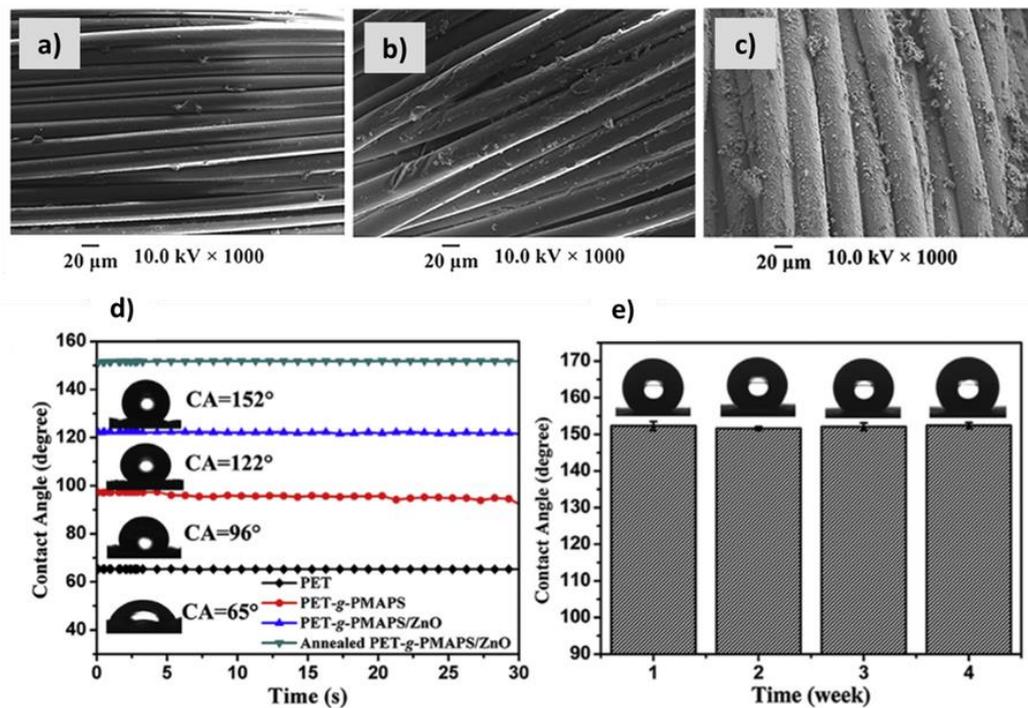


Figure 11. Surface morphology of pristine (a), polysiloxane coated (b) and polysiloxane-ZnO coated (c) polyester fibres; water contact angles (d) of pristine polyester (PET), polysiloxane coated polyester (PET-g-PMAPS), polysiloxane-ZnO coated polyester (PET-g-PMAPS/ZnO), polysiloxane-ZnO coated polyester after annealing (Annealed PET-g-PMAPS/ZnO); relationship between the water contact angles and storage time (e). Reprinted with permission from [106]. Copyrights 2018 Elsevier.

3.7. Electrical Conductivity

Dual semiconducting and piezoelectric properties enable ZnO to be used for the fabrication of wearable/textile electronics. Namely, the high piezo-photocatalytic efficiency enables ZnO to convert solar and mechanical energies into electricity, which is crucial for smart textiles [123–126]. Since this property is strongly dependent on structure and morphology, only ZnO grown on textile substrates in the form of a one-dimensional wurtzite nanocrystal structure, such as nanorods (NRs) and nanowires (NWs), can create a transparent conductive network with excellent electrical conductivity, low sheet resistance, and mechanical flexibility (Figure 12) [98,146–149]. To synthesize the ZnO NR array layer in situ on the surface of textile fibers, solvothermal [89], hydrothermal [98], ultrasonic irradiation [10], sol-gel [90,95], spin-coating [150], electro-deposition [9,111], and atomic layer deposition [93] methods were recently used.

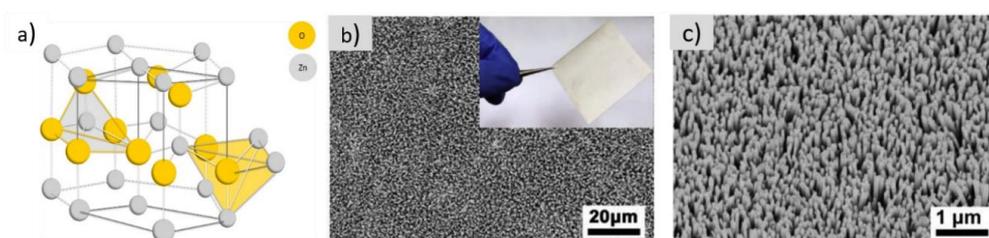


Figure 12. Hexagonal wurtzite crystal structure of ZnO (a) and ZnO nanorods grown on nylon fabric (b,c). Reprinted with permission from [98]. Copyrights 2019 Elsevier.

For wearable/flexible electronics, piezoelectric nanogenerators (PENGs) based on ZnO NRs arrays patterned silk and polyamide fabrics were developed [98,111]. The excellent coupled piezoelectric and semiconducting properties of the ZnO NRs enabled the textile substrates to harvest mechanical energy from human bodies and act as the mechanical sensing and monitoring devices (Figure 13).

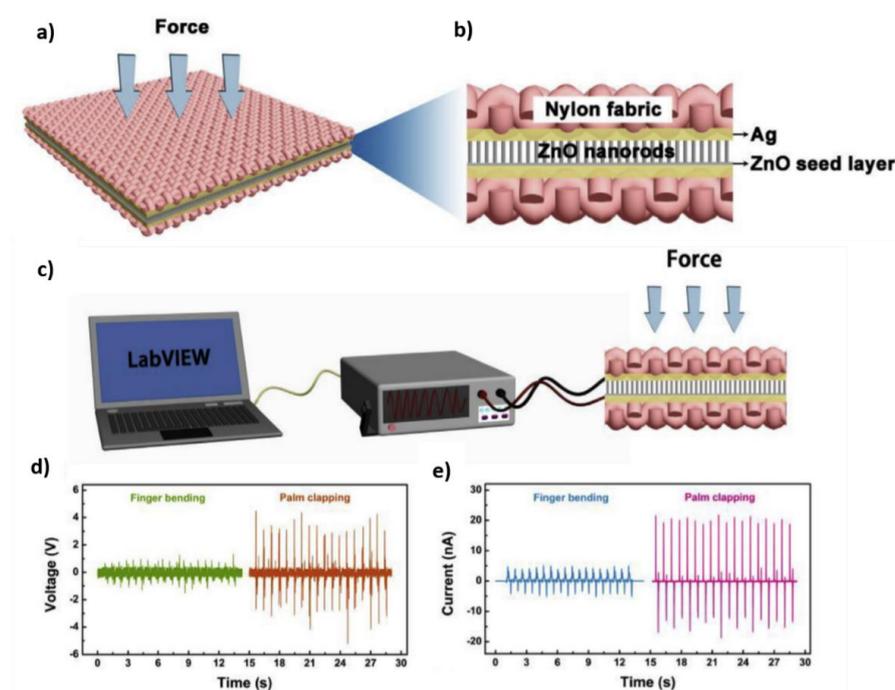


Figure 13. The structure of ZnO nanorods patterned textile based piezoelectric nanogenerator (PENG) (a); schematic diagram of PENG construction (b); schematic diagram of test system (c); output voltages (d) and currents (e) by finger bending and palm clapping. Reprinted with permission from [98]. Copyrights 2019 Elsevier.

To enhance the electrical conductivity, appropriate external dopants were also used in combination with ZnO. To this end, ZnO-benzene-1,4-diol superlattice coatings were deposited on a cotton surface with a predeposited aluminum oxide seed layer [93]; a cotton surface was coated with Sb-ZnO or Ag-ZnO nanocomposites [89]; keratin/poly(lactic acid) fibers were coated with ZnO-graphene quantum dots [150]; ZnO NRs were synthesized in situ on the surface of Ag-coated flax [95] and polyamide [98] fabrics; ZnO NRs were grown in situ on reduced graphene oxide [111] and gold [9] layers previously prepared on silk fabrics; finally, Mn-doped porous dodecahedral or star-shaped ZnO was applied to decorate carbon nanofibers [151]. To provide simultaneous electrical conductivity and hydrophobicity to cotton fibers, ZnO was applied in combination with tetradecanoic acid [81].

4. Conclusions and Future Perspectives

This paper reviews the most recent studies concerning ZnO-functionalized textiles. According to the literature, the most promising results were obtained in the production of textiles with photocatalytic self-cleaning, antimicrobial, UV-protective, flame-retardant, hydrophobic, thermo-insulating, and electrically conductive properties as well as textiles exhibiting moisture management. In some studies, in addition to the excellent performance of ZnO, a high durability of the functionalized material was obtained. These results confirm that ZnO is one of the most promising materials for the development of high-performance textile products and will therefore be intensively investigated in the future.

Future research in the field of ZnO-modified textile fibers will certainly follow the main directions in the fabrication of green, multifunctional, and smart textiles. Accordingly, investigations into new green procedures of ZnO synthesis in the presence of textile fibers with the use of environmentally friendly and nontoxic reagents, which will be able to control the chemical structure and morphological properties of ZnO NPs, will be of great importance. Other related studies should focus on improving the photocatalytic activity of ZnO NPs by decreasing their particle size and increasing their specific surface area, as well as on improving the visible light response and inhibiting the recombination of photogenerated h_{VB}^+ and e_{CB}^- by incorporating appropriate external dopants, coupling NPs with other semiconductors or modifying NPs with carbon materials. Among these research interests, the fabrication of different heterojunctions or composites with ZnO to broaden the light absorption region and facilitate the separation and transfer of photocarriers will be of great importance. In the development of wearable electronics, the enhancement of the piezo-photocatalytic activity of ZnO NRs by controlling the structure grown on conducting textile substrates will be crucial.

Along with the functional properties, the toxicity of ZnO, and the impact of the use of ZnO-modified textile fibers on human health and the environment will also play an important role in this field of research.

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Conflicts of Interest: The authors declare no conflict of interest.

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