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# Sol-Gel Applications in Textile Finishing Processes

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Additional information is available at the end of the chapter

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## Abstract

In the chapter, sol-gel applications in textile finishing process such as flame retardancy, water, oil repellency, ultraviolet (UV) protection, self-cleaning, and antibacterial and anti-wrinkle processes are reviewed. Sol-gel technology is well known in materials, metallurgy, ceramic, and glass industry since 1960 and has been researched in textile industry in the last decade. Sol-gel technology has some advantages when compared to conventional textile finishing process. Sol-gel technology, which is a method applied to the inorganic metal alkoxide or metal salts to organic textile materials, could impart the high, durable activity and multifunctional properties to different textile materials in the same bath at one step using low concentration of precursors. In addition, sol-gel technology presents alternatively economical, ecological, and environmental friendly process due to one-step application, using low concentration of chemicals, nonhalogenated chemicals, and nonformaldehyde release when compared to conventional processes.

**Keywords:** sol-gel, textile, cotton, polyester, flame retardancy, water, oil repellency, antibacterial, antiwrinkle, UV protection, self-cleaning, photocatalytic, multifunctional, silica, titania, silver, trialkoxy silanes

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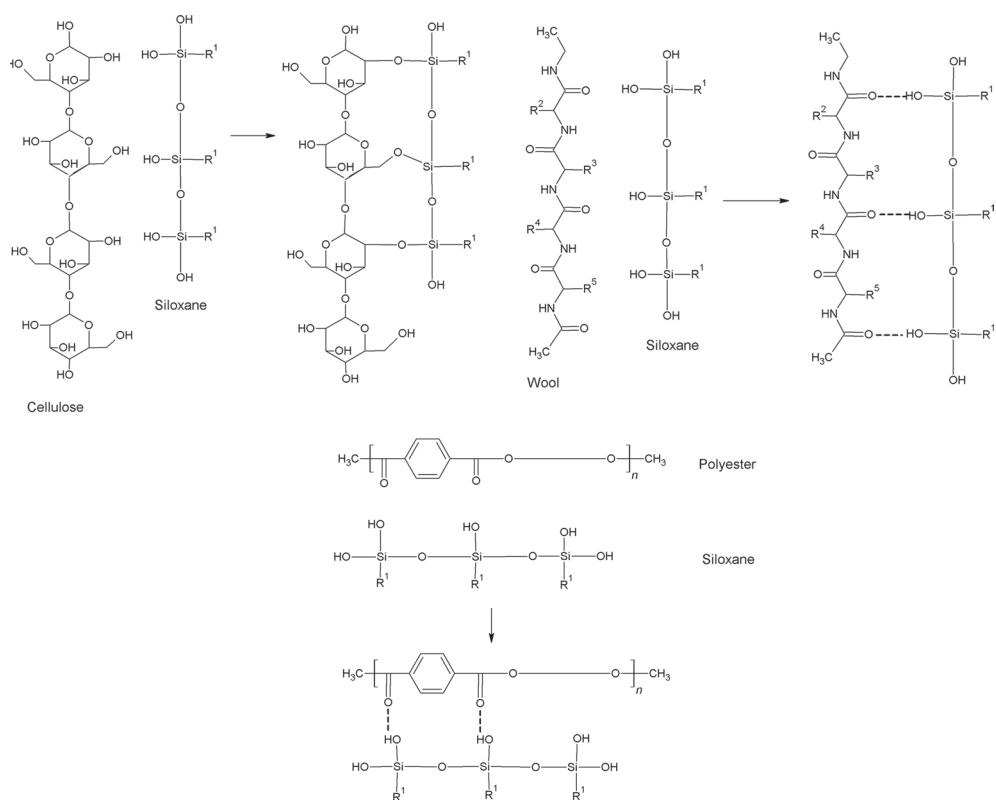
## 1. Introduction

Functional properties can be given to textile materials by textile finishing processes. The use of sol-gel technology in textile finishing processes has taken very much attention in the last decades. Sol-gel technology has advantages such as less chemical usage, low-temperature treatment, easy application and treatment facilities in textile mills, and no requirement for special equipment [1, 2].

Moreover, sol-gel technology makes enabling multifunctional properties in one step which is not possible by conventional textile finishing methods because of incompatibility of chemical materials. In conventional textile finishing methods, the treatments have

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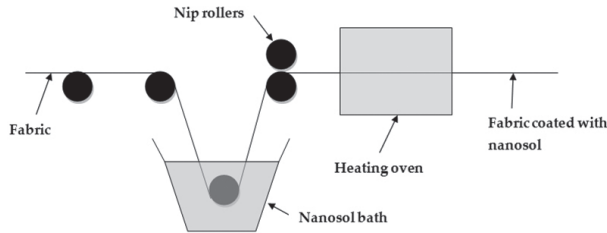
separately been applied in several steps to obtain functional properties and these multi-step processes increase the costs of the textile mills. Therefore, enabling multifunctional properties in one step is a challenging process to overcome in terms of decreasing water and energy consumption and costs. On the other hand, besides the various advantages, sol-gel technology has a disadvantage of high costs of precursor materials used in sol-gel technology. In sol-gel technology, metal alkoxides and metallic salts have been used as precursors and by the catalytic effect of an acid or alkali, hydrolysis and condensation reactions occur [1, 2]. The reactions of precursor materials with textile surfaces are given in **Figure 1**. Nanosols can be applied to fabric by pad-dry-cure process. Application steps of nanosols to textiles are illustrated in **Figure 2**.



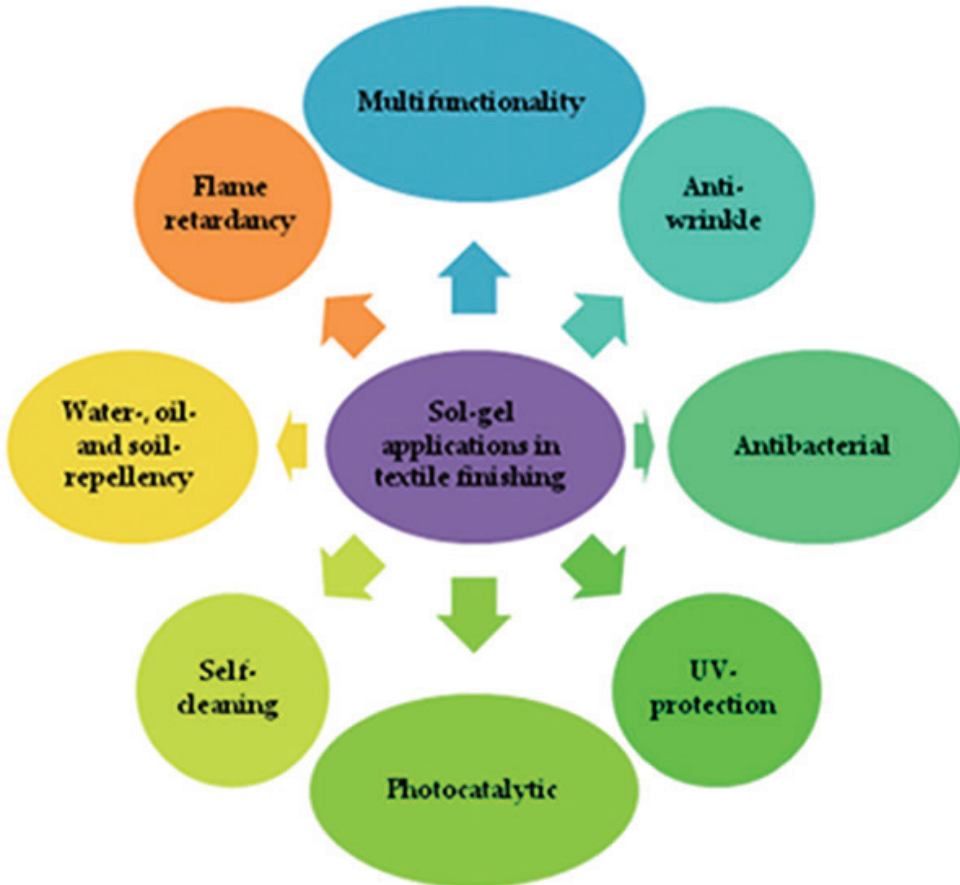
**Figure 1.** The reactions of precursor materials with some textile surfaces.

By sol-gel technology, various functional properties can be given to textile materials or some properties that already exist can be developed in one or more steps. The use of sol-gel technology in textile industry for flame retardancy, antimosquito [3], water or oil repellency [4], antibacterial activity [5], antiwrinkle, antcrease, durable press or easy-care effect [6], ultraviolet (UV) protection, self-cleaning or soil repellency, photocatalytic activity [7], development in

fastness behavior, abrasion resistance, tensile and thermal properties, and in microencapsulation process have been studied in detail. In **Figure 3**, the application areas of sol-gel technology in textile finishing processes have been demonstrated. The aim of this review is to introduce the usage possibilities of sol-gel technology in textile applications.



**Figure 2.** Application steps of nanosols to textiles.



**Figure 3.** Application areas of sol-gel technology in textile finishing process.

## 2. Textile finishing applications by sol-gel method

### 2.1. Flame retardancy finishing

There has been a significant importance to improve flame retardancy properties of textile materials for hindering the fire damages because they are primary combustion sources in a fire. Chloride-, bromide-, phosphorus-, antimony-, and boron-based materials have been used for textile materials to give flame-retardant activity. In addition, the combination of these chemical materials (e.g., antimony-halogen or phosphorus-nitrogen) can also be used due to their synergistic effect. By sol-gel method, high chemical concentration (nearly 300–500 g/l) used in conventional methods can be significantly decreased or by the use of chemical materials without halogen, ecological, and economical flame-retardant activity can be obtained. Alongi and Malucelli [8] reported the detailed review of flame retardancy treatment of cotton fabrics by sol-gel process until 2015.

Grancaric et al. [9] studied the flame retardancy properties of cotton fabric by using diethylphosphatoethyltriethoxysilane (DPTES) as a sol-gel precursor together with monoethanolamine (MEA) as a neutralizer for acidic conditions of DPTES with or without the addition of tetraethoxysilane (TEOS) and 3-glycidoxypropyltriethoxysilane (GPTES) to increase the durability of finishing process. Their results showed that the cotton samples were in self-extinguished classification and had the LOI (limited oxygen index) value of 29. It was observed that the amount of residue of treated fabrics at  $T_{\max}$  compared with that of untreated fabric increased from 37.5 to 60% and the total heat release (THR) of treated fabrics with respect to untreated fabric decreased from 12 to 4.2 kJ/g. Thermal shielding effect of silica phases and char-forming by synergistic effect of phosphorous and nitrogen were attributed to be the causes of flame retardancy and thermal behavior of cotton samples. However, the LOI values of fabric samples reduced from 29 to 21 after one washing process [9].

Boukhriss et al. [11] investigated the flame retardancy and water repellency properties of cotton fabric treated with sols containing 1-methylimidazolium chloride propyltriethoxysilane (MCPTS) and 1-pyridinium chloride propyltriethoxysilane (PCPTS) salts synthesized. The results indicated that their flame retardancy and water repellency properties improved. Water uptake values with dip test with regard to untreated fabric decreased from 340 to 50%. The weight of residue of treated fabric samples regarding untreated fabric increased from 1 to 93% which is higher than the test results (48%) reported by Alongi et al. [10]. The fabric samples lost their flame retardancy properties after three washing process and entirely burnt [11].

Kappes et al. [12] coated the cotton, PET fabric, and polyester/cotton blends with (3-trimethoxysilylpropyl)diethylenetriamine (TRIAMO) by sol-gel process. Then, coated and uncoated fabrics were treated with phenylphosphonic acid. Hole formation, flame spread to upper or side edges, and burning time after flame application of samples were evaluated by surface and edge-ignition tests according to EN ISO 15025. The two-step treatment of cotton and blend fabrics improved their flame retardancy properties and burning and hole formation after flame treatment of the samples were not observed while cotton fabrics treated with only sol-gel and only phenylphosphonic acid treatment showed burning and hole formation. Polyester fabric samples displayed hole formation and burning after flame treatment, but flame did not reach the upper

or side edges for the fabric samples treated with two-step process and only phenylphosphonic acid due to recede of fabric from flame. Sol-gel treatment gave rise to prolong burning time after flame treatment and to prevent fabrics receding from flame that is known to be undesired. Peak heat release rate (pHRR) of all fabric samples treated with two- and one-step process decreased compared with untreated fabric. The total heat evolved (THE) of polyester fabric slightly increased, while these of cotton and blend fabrics slightly decreased [12].

Zhang et al. [13] treated silk fabric to give flame retardancy by using TEOS and boric acid by means of sol-gel process. They used 1,2,3,4-butanetetracarboxylic acid (BTCA) as crosslinking agent to improve durability of flame retardancy properties of the fabric samples. The results showed that LOI values of silk fabric containing P, N, or S with inherent difficult flammability increased from 25.3 to 32.3% by treatment with BTCA solution and then nanosol-containing boric acid as a flame-retardant agent. They found that the flame retardancy of silk fabric was durable to even 30 washing cycles. They demonstrated that the pHRR, temperature at pHRR, the total heat release (THR), weight loss, and the density of released smoke of the fabric samples compared to control fabric decreased as indicating low flammability [13].

Šehić et al. [14] investigated the synergistic effect of the presence of Si- and P- on polyamide 6 fabric surface treated by sol-gel process using 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO)-modified vinyltriethoxysilane (VTS) and tetraethoxysilane (TEOS) as precursors. The increase in the percentage of residue at  $T_{\max}$  and the decrease in  $T_{\text{onset}}$  were attributed to promoting char formation. The heat release capacity (HRC), pHRR, and THR of fabric samples were found to be reduced. Ignition of cotton under burning samples, after-flame, and after-glow times were analyzed by vertical flame-spread test, DIN 53906. The combination of Si and P and the presence of only Si contributed to no dripping, no ignition of cotton substrate under burning sample, longer after-flame time, and no after-glow. Synergistic effectiveness values of samples proved the synergistic activity of Si and P in condensed phase. **Figure 4** shows the combustion cycle of polymer [15].

Deh et al. [16] studied the synergistic effect of N and Si added to phosphorous compounds on flame retardancy of cotton fabric treated by phosphorylation using phosphoric acid and urea and then by sol-gel process using TEOS as a precursor. They found that the cotton fabrics treated by phosphorylation and sol-gel process had high LOI values (>25%) with durability to 10 washing processes. The presence of Si together with N and P on cotton fabric contributed to developing the formation of char acting as a thermal-insulating barrier and to decrease the number of byproducts which are not promoting the dehydration process, is requested flame retardant activity. Dehydration of cotton at low temperature was observed to support char formation and synergistic effect reduced the formation of levoglucosan whose decomposition generates flammable gases and product after pyrolysis. **Figure 5** illustrates the thermal degradation of cotton with heating [16].

Liu et al. [17] synthesized di-(triethoxysilylpropyl) phenylphosphamide (PPD-PTES) by reaction of 3-aminopropyl triethoxysilane (APTES) and phenylphosphonic dichloride (PPDC) and then treated cotton fabric with PPD-PTES for 5, 10, 20, and 30 min of immersion times by sol-gel process. As a result of vertical burning test, the fabric treated with PPD-PTES containing P-, Si- and N-elements self-extinguished as ignition source was found to be withdrawn and minimum char area left was observed on the fabric treated with 30 min of immersion

time. As long as immersion times were increased, char residue increased while  $T_{max}$ , pHRR, and THR decreased. Nevertheless, they found that the absorbance intensity of  $CO_2$ , which is one of the non-flammable gases, did not significantly change in treated fabrics compared to that of untreated fabric while their intensity of C=O and C-O-C groups, which are found

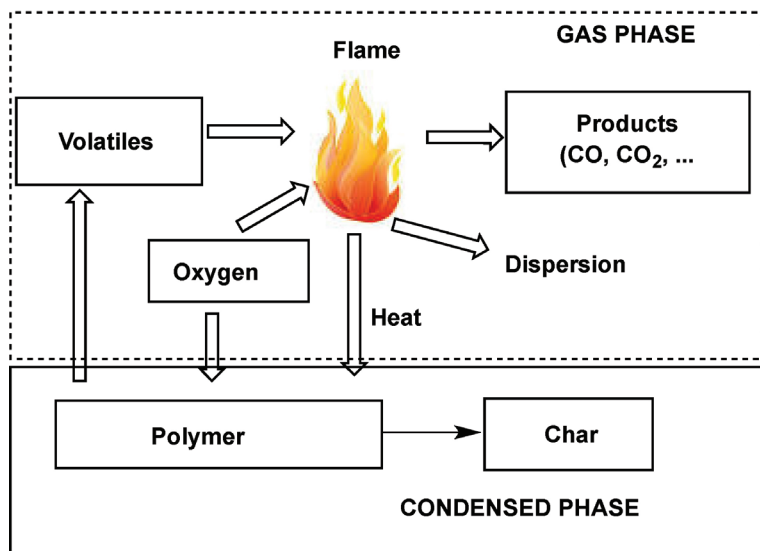


Figure 4. Schematic illustration of combustion cycle of polymer [15].

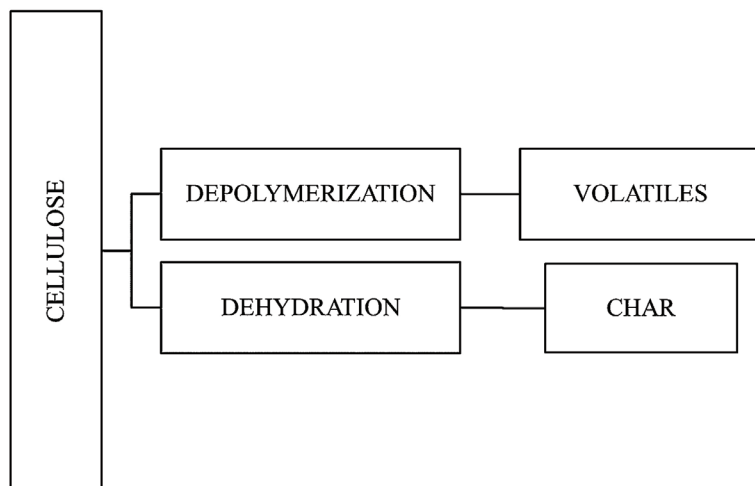


Figure 5. Schematic illustration of the thermal degradation of cotton with heating. Copyright 2015, Royal Society of Chemistry [8].

in flammable gases and compounds, decreased by virtue of Fourier transform infrared spectroscopy (FTIR) analysis [17].

Ren et al. [18] investigated the flame retardancy properties of polyacrylonitrile (PAN) fabric treated by one-step sol-gel process using TEOS and polyphosphoric acid (PPA). The increase at their LOI and char residue to, respectively, 30.1 and 69.48% and the decrease at their THR, pHRR, the fire growth rate index (FIGRA), average mass loss rate (amLR), the peak of smoke production rate (pSPR), and total smoke production (TSP) as a result of cone calorimetry test [18] were determined.

Aksit et al. [19] proved the synergistic effect of Si, P, and N on flame-retardant cotton fabrics treated with (3-aminopropyl) triethoxysilane as N- and Si-source, (3-glycidyoxypropyl) trimethoxysilane as Si-source, and guanidine phosphate as P-source by sol-gel technique. They determined that the fabric samples had 45.7% LOI values [19].

## 2.2. Water or oil repellency finish (hydrophobic effect)

Hydrophobic effect can be given to textile materials by decreasing the surface tension of textile material against the liquid [1]. In conventional methods, fluorocarbon components have been used for this purpose. However, sol-gel technology as an alternative method has also been studied because of the harmful effects of fluorocarbon components to the environment. Many researches have been conducted about the use of modified silane precursors by sol-gel technology in order to enable hydrophobic effect on textile materials. It was aimed to gain surface roughness together with creating low-surface energy on substrate surface based on the theory of Wenzel or Cassie and Baxter [20–22] in order to produce superhydrophobic materials in many researches. Moreover, the durability to abrasion, washing and UV exposure, and so on of their superhydrophobic properties for especially outdoor materials were maintained as a significant challenge. Schematic illustration of hydrophilicity-hydrophobicity properties of substrate as contact angles is given in Figure 6.

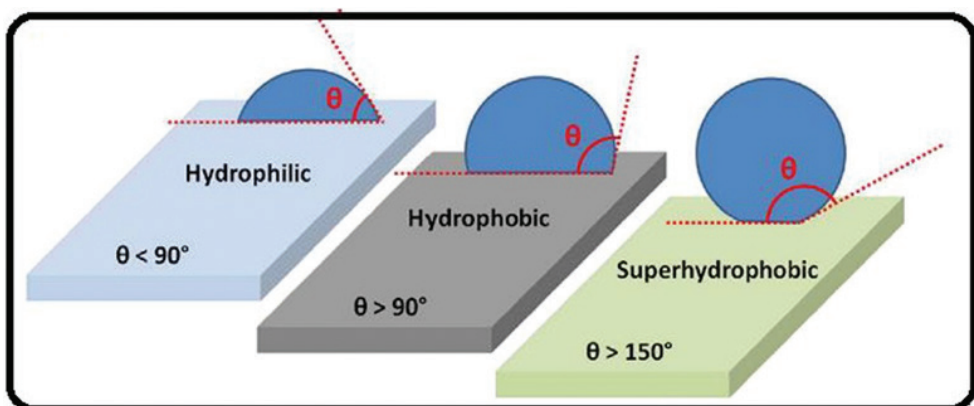


Figure 6. Schematic illustration of hydrophilicity-hydrophobicity properties of substrate as contact angles [23].

Zhao et al. [24] treated the cotton, cotton/polyester, and polyester fabric by using nanosol containing silica nanoparticles, (3-aminopropyl) triethoxysilane (APTES, 99%), and hexadecyltrimethoxysilane (HDTMS) by spray process. They achieved the production of fabrics with contact angle (CA)  $>150^\circ$  and hysteresis  $<10^\circ$  required to gain superhydrophobic properties to fabric. The increasing amount of HDMS or silica nanoparticles led to higher contact angle and lower-surface energy than that of uncoated fabrics. Surface roughness values measured by atomic force microscopy (AFM) were found to increase for cotton and cotton/polyester fabric compared to uncoated fabrics. The cotton and polyester fabric did not lose their superhydrophobic properties after five washing processes. In addition, the fabrics kept their hydrophobic properties even after 600 abrasion cycles [24].

Xue et al. [25] coated polyester fabric with polymer paste containing silica nanoparticles and polydimethylsiloxane (PDMS) by spread coating method and then posttreated by nanosols containing TEOS and cetyltrimethoxysilane (CTMS). They determined the hydrophobic properties (CA  $>90^\circ$ ) of fabrics coated with PDMS-silica nanoparticles and superhydrophobic properties (162.5 of CA, 5.4 of sliding angle (SA)) of fabric posttreated by TEOS and CTMS. From the study, it was observed that water pressure resistance properties (38.6 kPa) of fabrics developed, their CAs a little decreased, and their SAs slightly increased as the number of coatings increased. Their contact angles and water resistance properties did not demonstrate significant change after basic and acidic treatment with different pH values or after UV-light exposure. However, water pressure resistance of fabrics decreased to 10 kPa after abrasion test for even 10 cycles because of the insufficient binding force between coating layer and fabric [25].

Hao et al. [26] investigated polymer nanocomposites (PNs) synthesized by *in situ* sol-gel process to achieve multifunctional properties of cotton fabrics. Crosslinked polysiloxane (CLPS) with aminopropyltriethoxysilane (APTES) and then obtained APTES-CLPS with silica sols (CLPS-SiO<sub>2</sub>) were treated by *in situ* sol-gel process. The cotton fabric was immersed in APTES-CLPS or CLPS-SiO<sub>2</sub> solutions in ethyl acetate and padded, dried, and cured. They concluded that the fabric samples treated with CLPS-SiO<sub>2</sub> had better thermal stability with lower weight loss in TGA analysis, higher the root-mean-square roughness (4.528 nm) than that treated with APTES-CLPS due to the presence of silica nanoparticles. The contact angles of the fabric reached  $158^\circ$  and they did not lose their superhydrophobic properties until 10 washing cycles [26].

Zhang et al. [27] produced an oligomer of hexadecyltriethoxysilane (HD-oligomer) (HDTES) and HDTES-modified silica nanoparticles (HD-silica) by Stöber method based on sol-gel process. In the second step, HD-silica/HD-oligomer nanocomposites were prepared by adding HDTES, TEOS, ethanol, water, and ammonia to HD-silica and HD-oligomer. Subsequently, wool fabrics were immersed in nanocomposite solution in toluene and triethylamine as catalyst and exposed to ultrasonication to produce superhydrophobic wool fabric with excellent abrasion resistance, excellent chemical and environmental stability, excellent mechanical resistance for adhesion of double side tape and scratching with a scalpel, and good washing durability for 10 washing cycles [27].

Vasiljević et al. [28] studied the improvement of water repellency properties of the cotton fabric by first treating with oxygen plasma and then with 1H,1H,2H,2H-perfluorooctyltriethoxysilane (SiF) in ethanol by sol-gel process. The fabrics pretreated by plasma for 20 s at an operating current of 0.3 A resulted in nearly  $150^\circ$  of contact angle and  $24^\circ$  of sliding angle [28].



Przybylak et al. [29] previously impregnated cotton fabric with octaanion solution synthesized by using TEOS, tetramethylammonium hydroxide, methanol, and water and squeezed, dried, and subsequently impregnated with bifunctional silsesquioxanes containing octafluoropentylpropyl groups and trimethoxysilylethyl synthesized. Squeezed, dried, and cured fabric samples were found to have superhydrophobic properties with  $151^\circ$  of contact angle and durability against 10 washing cycles [29].

Xue et al. observed that the fabrics treated with nanosols containing tetrabutyl titanate and then with 1H, 1H, 2H, 2H-perfluorodecyltrichlorosilane in toluene and/or stearic acid in acetone and cured at  $110^\circ\text{C}$  for 1 h had superhydrophobic properties and good UV-protection properties [30].

Onar and Mete [31] investigated water repellency properties of the cotton fabric treated with two-step process as surface roughening with ZnO,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ , and  $\text{ZrO}_2$  nanoparticles and hydrophobic modification with long-chain alkyl silanes (hexadecyltrimethoxy silane). From the study, it was observed that the cotton fabric treated with  $\text{Al}_2\text{O}_3$  nanoparticles and HDMS had superhydrophobic properties and all fabric samples treated with nanoparticles and HDMS maintained their hydrophobic properties after washing process [31].

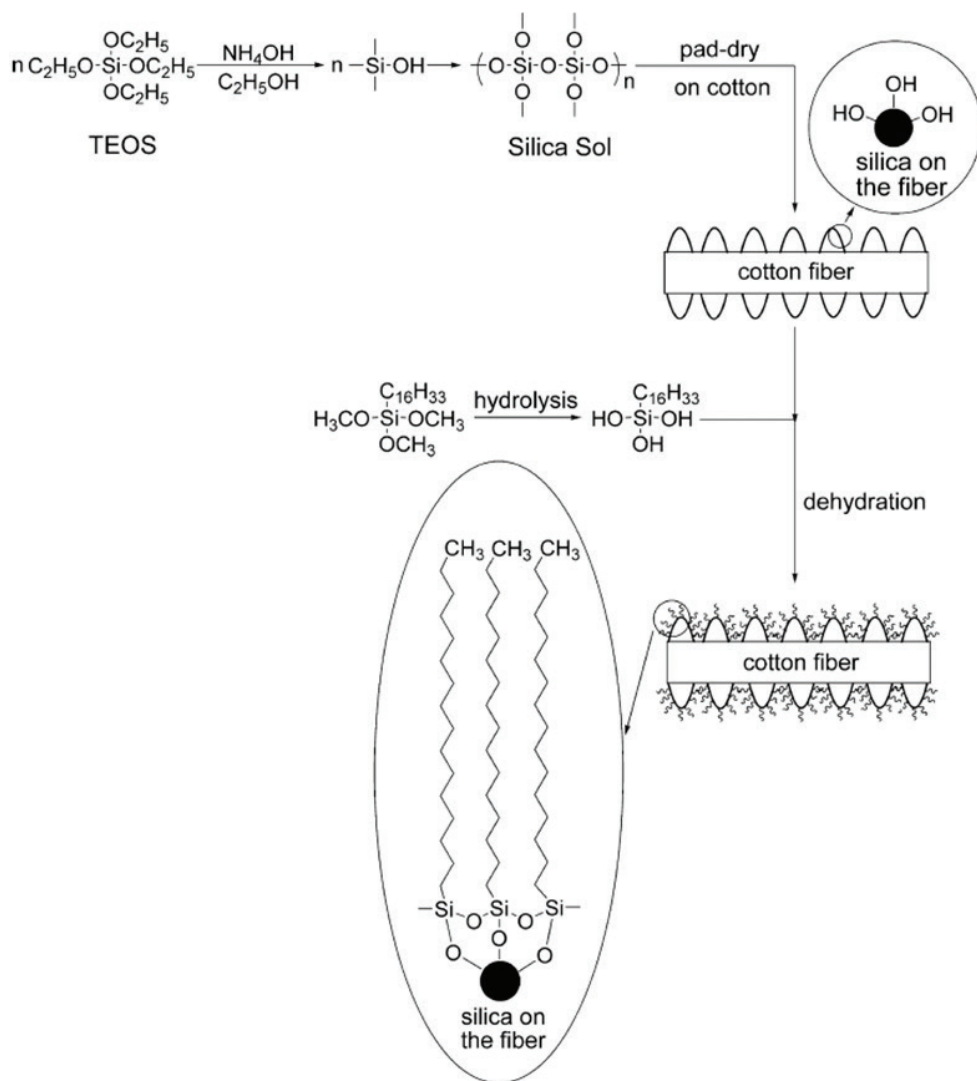
Onar et al. [32] purposed to achieve the production of water-repellent cotton fabric by sol-gel process using tridecafluorooctyltriethoxysilane (FTES), hexadecyltrimethoxysilane (HDTMS), glycidylpropyltriethoxysilane (GLYE), phenyltriethoxysilane (FES), vinyltrimethoxysilane (VTEO), 3-aminopropyl trimethoxysilane (AMMO), titanium (IV) isopropoxide (TIPT), and zirconium (IV) acetylacetonate (ZrAc) as precursors. They found that the treatment with HDTMS under acidic and alkaline conditions or FTES under alkaline conditions led to provide superhydrophobicity on cotton fabric. The fabric samples treated with FES, VTEO, TIPT, and GLYE precursors lost their hydrophobic properties after washing process, while the fabrics treated with HDMS, FTES, AMMO, and ZrAc precursors preserved the hydrophobic properties after washing process [32].

Colleoni et al. [33] treated the cotton and polyester fabric with octyltriethoxysilane (OTES) together with melamine-based crosslinking agent. They revealed that the cotton and polyester fabrics had good hydrophobic properties with, respectively,  $130^\circ$  and  $150^\circ$  of contact angle values [33].

Gao et al. [34] researched the hydrophobic properties of the cotton and polyester fabric treated with TEOS and then HDTMS by sol-gel process (**Figure 7**). The process imparted highly hydrophobic properties to the cotton ( $155^\circ$  CA) and polyester fabric ( $143^\circ$  CA) with durability till 30 washing processes and a slight reduction of their tensile strength ( $<10\%$ ) [34].

Teli and Annaldewar [35] studied the water repellency and UV-protection properties of cotton fabric dip coated with silica nanoparticles prepared by sol-gel process and subsequently impregnated with zinc nitrate hexahydrate in hexamine and then hydrophobically modified with ethanolic sodium stearate solution. As a result of that, the fabric samples imparted excellent UV protection with +50 UPF and superhydrophobic properties. The fabric samples had still excellent UV protection and hydrophobic properties after 10 washing cycles [35].

de Ferri et al. [36] coated the silk fabric with nanosols composed of 3-glycidoxypropyltrimethoxysilane (GLYMO) and DynF8815 with acid catalyst by sol-gel process. The treatment caused the high abrasion resistance to 8500 cycle, high contact angle (nearly  $123^\circ$  CA) to four oils, and hydrophobic properties with  $148^\circ$  of contact angle. The washing durability of oil and water repellency of the fabric samples was also analyzed. It was observed that the fabric had still hydrophobic ( $130^\circ$  CA) and oleophobic properties ( $100^\circ$  of CA) after, respectively, 15 washing and 5 washing cycles [36].



**Figure 7.** The reaction of cotton fibers with previous TEOS and the HDTMS. Copyright 2009 American Chemical Society [34].

Vasiljević et al. [37] aimed to produce highly oleophobic and superhydrophobic cotton fabric by three-step sol-gel process. The first step was the treatment of cotton fabric with silica nanoparticles with different average particle sizes ( $50 \pm 15$ ,  $230 \pm 20$ , and  $780 \pm 30$  nm) prepared according to Stöber method based on sol-gel process; the second step was the application of the *in situ* growth of polysiloxane layer on cotton fabric in basic TEOS solution, and the third step was the padding with a fluoroalkyl functional water-borne oligosiloxane (FAS, Dynasylan F 8815). They obtained fabric samples with low sliding angle ( $2^\circ$ ), high oleophobicity (6), and superhydrophobicity durable to 10 washing cycles [37].

### 2.3. UV protection, self-cleaning, and soil repellency (photocatalytic effect)

Long-time exposure to UV light may result in early skin aging, allergy, sun burns, or even skin cancer in especially fair-skinned human. Various chemical agents such as benzophenone derivatives, ortho-hydroxy phenyl, and diphenyl triazine derivatives as organic UV absorbers and  $\text{TiO}_2$  and ZnO particles as inorganic UV absorbers have been used [38]. Alternatively, UV protection can be obtained by sol-gel method with generally  $\text{TiO}_2$  and ZnO nanoparticles incorporated to textile materials.  $\text{TiO}_2$  nanoparticles provide self-cleaning as well as UV protection by means of photocatalytic reaction. Anatase phase of  $\text{TiO}_2$  nanoparticles, which is one of three phases of  $\text{TiO}_2$  (anatase, brookite, and rutile), has photocatalytic activity. The formation of anatase phase at low temperature on textile material coated with titania nanosols is a big challenge because textile materials cannot endure high temperature [39]. Researchers are investigating some solutions such as steaming process [40, 41], hydrothermal process [42], or solvothermal process [43] to produce anatase crystals at low temperature so far.

Photocatalytic reaction leads to the decomposition of organic and inorganic pollutants under the ultraviolet light [43]. In addition, organic components such as microorganisms in textile surfaces and dyestuff residues in textile wastewater can also be decomposed by photocatalytic effect. By this way, photocatalytic effect ensures not only UV protection and self-cleaning but also antibacterial effect and ecological and economical wastewater treatment [43, 44]. **Figure 8** illustrates the self-cleaning properties of cotton fabric treated with titania nanosols.

Liu et al. [46] investigated the self-cleaning and UV-protection properties of raw wool fabric and Kroy-process wool fabric (removed scales with chlorination process) treated with polycarboxylic acid such as 1,2,3,4-butanetetracarboxylic acid (BTCA), citric acid (CA) as crosslinking agent and then nanosols prepared by using titanium (IV) butoxide (TBT) and TEOS as precursors. The self-cleaning properties of the fabric samples were evaluated by photo-induced decomposition test of methylene blue. Self-cleaning properties of raw wool fabric were found to be better than that of Kroy-process wool fabric, while UPF values of raw material (356) were lower than that of Kroy-process wool fabric (992) [46].

Dhineshabu et al. [47] studied the UV-protection, antibacterial, and flame retardancy properties of cotton fabric treated with colloidal sols containing TEOS as precursor and polyethylene glycol (PEG) as stabilizer and  $\text{TiO}_2$  nanoparticles produced by sol-gel process. They found that UPF values of the fabric samples were greater than 50 UPF corresponding to excellent UV protection after even 10 washing cycles. According to ASTM D1230 standard, their results

showed that the total burning time and residual weight after burning of the fabric samples were higher than that of untreated fabric according to the flame test at 45° angle. Thus, their burning performance improved after the treatment and nearly 70% of this performance was preserved after 10 washing cycles. The study revealed that the fabric samples had good antibacterial activity against *Staphylococcus aureus* and *Escherichia coli* with, respectively, 18 and 15 mm diameter of inhibition zone because of the reactive oxygen species (ROS) generation mechanism of TiO<sub>2</sub> nanoparticles [47].

Rilda et al. [48] previously produced TiO<sub>2</sub>-SiO<sub>2</sub> powder by using titanium isopropoxide and TEOS as precursors with different molar ratios, diethanol amine (DEA) as stabilizer, isopropanol as solvent, and chitosan in acetic acid as dispersing agent by sol-gel process. The fabric samples coated with acrylic acid binder were immersed to TiO<sub>2</sub>-SiO<sub>2</sub> suspension containing the powder, surfactant, and isopropanol and then coated with the suspension to ensure homogeneous coating by spin-coating machine. Falling at absorbance of methylene blue under UV irradiation for 120 min was attributed to self-cleaning properties of fabrics treated with TiO<sub>2</sub>-SiO<sub>2</sub> powders with especially 1:2 molar ratios [48]. The discoloration of wine stain on cotton fabric treated with TiO<sub>2</sub>-SiO<sub>2</sub> colloidal solution increased exposure times under solar light simulator for 0, 4, 8, and 24 h [49].

Wang et al. [39] treated the cotton fabric with TiO<sub>2</sub> acidic hydrosol prepared with titanium (IV) butoxide as precursor by dip-pad-steam process. Photocatalytic activity of TiO<sub>2</sub> prepared by low-temperature steaming process was higher than that of TiO<sub>2</sub> dried at 150°C. They stated that the fabric samples had excellent durable self-cleaning and UV-protection properties. The crystallinity and photocatalytic activity of TiO<sub>2</sub> improved with the increase of the water content in TiO<sub>2</sub> hydrosol [39].

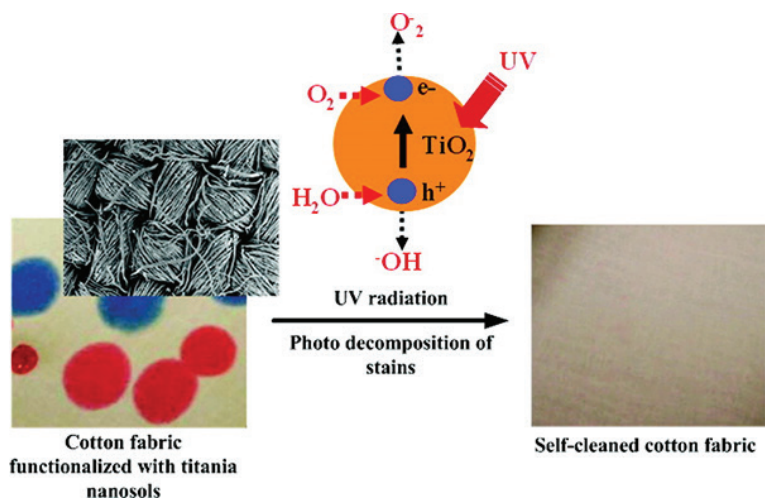


Figure 8. Self-cleaning properties of cotton fabric treated with titania nanosols. Copyright 2009 American Chemical Society [45].

Shaban et al. [50] investigated the self-cleaning properties of cotton fabrics coated with ZnO nanoparticles or ZnO solution containing zinc acetate dihydrate as precursor, 2-methoxyethanol as solvent, and monoethanolamine as stabilizer by sol-gel spin-coating process. Their results indicated that ZnO nanoparticles had photocatalytic activity especially under UV irradiation due to their high band-gap energy and the methyl orange dye on cotton fabrics coated with ZnO nanoparticles or ZnO solution degraded under sunlight and 200-W lamp illumination [50].

Noorian et al. [51] prepared precursor solutions based on copper sulfate and/or zinc chlorite containing folic acid, NaOH, and water by in situ synthesis. They immersed the cotton fabric in the solutions, dried and washed. They stated that the combination of ZnO and Cu<sub>2</sub>O and the addition of folic acid improved the UV protection and anticrease properties of the fabric samples with, respectively, 87.31% enhanced UVP and 100.75° of crease recovery angle [51].

#### 2.4. Antibacterial and antimicrobial activity

Antibacterial and antimicrobial activities are important for sport, outdoor, home, automotive, and medical textiles and for garments like hosiery and underwear. For antibacterial and antimicrobial textile finishing, various biocides like silver, triclosan, chitosan, quaternary ammonium salts, N-halamines, biguanide derivatives, synthetic dyes, and peroxyacids have been applied to textile surfaces [52–58]. Sol-gel technology as an alternative method can bring advantages like ecological treatment, less chemical use, low-temperature processing, low toxicity to human health, protection of inherent properties of textile material, and excellent washing and usage durability in this finishing process [52, 55, 59–61].

Different types of sol-gel systems are known to have antibacterial or antimicrobial effect. These systems are photoactive titania coatings with anatase modification and sol-gel coatings with embedded colloidal metal or metal compounds like silver, silver salts, copper compounds, zinc, or biocidal quaternary ammonium salts [5, 62–65]. On the other hand, these systems have several disadvantages like high operation temperatures to produce highly photoactive thin films and the use of strong acids to keep aqueous sols in the peptized state that cause destruction on textiles. In addition, titania coatings require UV radiation to perform antimicrobial or antibacterial activity [66–69]. Because of harmful effects of strong acids and high temperatures on textiles, in recent methods, organic solvents, for example, alcohol-based solutions that can be applied at low temperatures, were developed [5, 59, 60]. Simoncic et al. compiled the antibacterial activity of silver nanoparticles prepared by *in situ* method and colloidal solutions [70].

Poli et al. prepared zinc-based coatings by sol-gel method in neutral hydro-alcoholic medium and applied on cotton fabric to obtain antibacterial activity. They reported that this method was simple, cheap, reproducible, and less harmful when compared to conventional methods [5]. Other than titania-based sol-gel coatings, silica-based coatings are also under examination for antibacterial or antimicrobial effectiveness. To enable the antibacterial or antimicrobial effect, polycationic components are incorporated within the silica layer matrix and by this means positively charged polycationic components interact with negatively charged microbial cell membranes and damage their cellular functions [5, 61, 62, 63]. The use of these systems was researched in detail [5, 59–63, 67].

Mahlting et al. reported that the sol-gel processing exhibited the antimicrobial activity based on controlled release, contact action, or photocatalytic activity [71].

Rivero and Goicoechea presented the detailed review about improving antibacterial properties of textiles by sol-gel process [72].

Zhang et al. classified the application methods on cotton methods for colloidal suspensions of metal nanoparticles and precursor solutions of metal ions, respectively, titled as sol and solution to gain antimicrobial properties [52].

Mohamed et al. [73] prepared the colloidal solutions of silver nanoparticles (10–25 nm) synthesized by chemical reduction process using dextran as stabilizing and reducing agent and modified TEOS using ascorbic acid as scavenging agent and (3-aminopropyl)triethoxysilane (APTEOS) by using BTCA or vinyltriethoxysilane (VTEOS) and applied it to cotton fabrics by sol-gel process by dip coating. Silver nanoparticles modified with TEOS and VTEOS were cured with UV irradiation by means of a photoinitiator. They proved that the modified fabrics had antibacterial activity against *S. aureus* and *E. coli* with nearly 90% of bacterial reduction even after 20 washing cycles and thermo-regulating properties [73].

The combination of antibacterial biopolymers with titania and silica matrix is a different method and this method is observed to give ecological advantage and possessed antibacterial activity both in the presence or absence of light. Arik and Seventekin [55] studied chitosan/titania and chitosan/silica hybrid coatings in terms of antibacterial activity. Sol-gel method was used to prepare coating solutions and then solutions were applied to cotton fabric. From the study, hybrid coatings were found to have better antibacterial activity and washing durability than only chitosan, only titania, or only silica coatings [55].

### **2.5. Antiwrinkle, antcrease, durable press or easy-care effect**

Antiwrinkle, antcrease, durable press, or easy-care effect has been enabled by the fixation of the crosslinking agents to textile materials. Since dimethyloldihydroxyethylene urea (DMDHEU), which was used commonly in conventional antiwrinkle finishing treatments, releases free formaldehyde that is harmful to human health and environment and causes negative effects due to the acidic catalysts on tensile properties and whiteness index of textile materials, new alternative crosslinking agents and methods have been recently researched. By means of sol-gel technology, it is possible to enable antiwrinkle effect herewith good tensile and whiteness properties by the use of silane coupling agents [74, 75] like glycidoxy propyltrimethoxysilane as epoxy silanes, vinyltrimethoxysilane as vinyl silanes [76, 77], and tetraethoxysilane that do not release formaldehyde [78–80].

The main cause of the tensile strength loss and yellowing in conventional antiwrinkle finishing is the disruptive effect of the acidic catalyst on fibers at high temperatures. But in sol-gel method, metallic alkoxides and organic solvents are used in mild conditions that provide tensile strength and color retention [78–80].

In antiwrinkle sol-gel applications, mixed sols with conventional agents can also be applied and different combinations like DMEU/SiO<sub>2</sub> (dimethylolethyleneurea/silica), TEOS-TTB/

DMDHEU (tetra ethoxysilane-titanium (IV) n-butoxide/dimethylol dihydroxyethylene urea), BTCA-MAH/SiO<sub>2</sub> (1,2,3,4-butanetetracarboxylic acid-maleic anhydride/silica), BTCA-SHP/TEOS-GPTMS (1,2,3,4-butan tetracarboxylic acid-sodium hypophosphite/tetraethoxysilane-glycidylpropyloxytrimethoxysilane), DMDHEU/TEOS-GPTMS were evaluated for antiwrinkle finishing and their effects on physical properties of cotton in previous studies [75, 78–81]. These combinations were also found to have advantages in terms of abrasion resistance [75], heat resistance [78], and UV-light resistance [79] as well as antiwrinkle effect.

Schramm and Rinderer treated the cotton fabric with BTCA and/or nanosol containing GPTMS together with metal alkoxides such as aluminum isopropoxide (AIP), titanium tetra isopropoxide, and zircon tetrabutoxide (ZTB) or hydrophobic trialkoxysilanes such as methyltriethoxysilane (MTEOS), octyltriethoxysilane (OTEOS), or Dynasylan F8815 (fluoroalkyl functional oligosiloxane) to impart antiwrinkle properties. Treatment only with metal alkoxides led to no improvement of their anticrease properties, while treatment with GPTMS developed their anticrease properties. Moreover, the addition of AIP and OTEOS or MTEOS to GPTMS solution caused the increase of dry-crease recovery angle (DCRA) of the fabrics treated with the solution, respectively, from 263° to 289°, 290°, and 294°. Tensile properties of fabric treated with GPTMS solution decreased 10% when compared to that of untreated fabric. The addition of AIP solution to GPTMS solution brought on further 10% loss of their tensile strength, while the addition of OTEOS and MTEOS solutions to solution containing GPTMS and AIP induced to increasing 10% of the tensile strength with regard to tensile strength of fabrics treated with solution containing GPTMS and AIP. The treatment with GPTMS solution containing AIP or AIP together with F8815, MTEOS, and OTEOS solutions ensured the enhancement of contact angle values of the fabrics, respectively, from hydrophilic to 112°, 147°, 145°, and 136° corresponds to excellent hydrophobic properties. They also found that BTCA pretreatment improved the anticrease properties of the fabrics treated with GPTMS solution until 299° of DCRA and led to further loss of tensile strength of the fabric samples treated with GPTMS solution. BTCA pretreatment also did not cause significant change on contact angle values of the fabric samples-treated GPTMS together with metal alkoxides and/or hydrophobic trialkoxysilanes [22].

## 2.6. Multifunctional finishing

It is possible to give various functional properties to textile materials in one step by the combination of compatible inorganic precursors, thanks to sol-gel technology and the finishing like this is called multifunctional finishing. Multifunctional finishing has drawn much attention in the last decades with many studies on this subject. **Table 1** summarized some researches about various functionalities on textile materials treated with *in situ* sol-gel method and sols with nanoparticles [82–101].

Memon et al. treated polyester fabrics with titania-doped silica nanosols. It was observed that the antiwrinkle and UV-protection properties developed, while air permeability and whiteness index values decreased only slightly [102].

Onar and Mete developed water-oil-repellent and flame-retardant cotton fabrics by sol-gel method or knife-over-roll coating using a one-step treatment. The nanosol containing TEOS as a precursor, hexadecyltrimethoxysilane as a precursor with water-repellent properties, and

Sol preparation	Functionality	Textile	Precursor or NPs	Solvent	Catalyst	NP binding	Other chemicals/ function	Ref. no.
NP sols	Anti-UV, rubbing fastness	Co	TEOS, GPTMS	Ethanol, water	NH <sub>4</sub> OH	Polyacrylate/adhesive, NM-5263/thickening agent	Dodecanol/nonsurfactant template	[82]
NP sols	UPF, hydrophobicity, antibacterial, mechanical strength	Co/Pes (65/35, 20/80)	TiO <sub>2</sub> NP, ZnONP	Toluene	-	Acrylate-based binder	-	[83]
NP sols	Electroconductive, antibacterial	Co, CV	AgNO <sub>3</sub>	Ethanol	-	-	PVP/stabilizer, EG/reducing agent, NaCl/mediating agent	[84]
<i>In situ</i> sol-gel	Antibacterial, self-cleaning	Co	AgNO <sub>3</sub>	Water	-	Acrylic acid/plasma polymerization monomer	NaBH <sub>4</sub> /reducing agent	[85]
<i>In situ</i> sol-gel	UV-resistance, thermal properties	UHMWPE	Titanium butoxide	Ethanol, water	Acetic acid	-	MPTMS/graft polymerization monomer	[86]
<i>In situ</i> sol-gel	Antibacterial, antifungal, abrasion, pilling resistance	Co/PES	GPTMS, aluminum isopropoxide	Ethanol, water	HCl	PVA	Ag/CuNP/bioactive additive	[87]

Table 1. (continued).



Sol preparation	Functionality	Textile	Precursor or NPs	Solvent	Catalyst	Other chemicals/ function	Ref. No
<i>In situ</i> sol-gel	UV-protection, antibacterial, dyeing	Co	TIP	Ethanol, water	Acetic acid	CHPTMAC/cationization agent, dyes	[88]
<i>In situ</i> sol-gel	Surface bonding, carrier for chemicals / drugs / therapeutic	PES, Co	TEOS, APTES, AEAPTMS	Ethanol, water	-	Sodium silicate solution/ nucleation agent	[89]
<i>In situ</i> sol-gel	Flame retardancy	Silk	TEOS	Ethanol, water	-	Dimethyl phosphonate	[90]
<i>In situ</i> sol-gel	Thermal resistance, water-repellency	Co	PVS (with VTES)	Ethanol, water, toluene	HCl	-	[91]
<i>In situ</i> sol-gel	Antibacterial, self-cleaning	Co	Zinc nitrate	-	NaOH	<i>Tragacanth gum</i> /reducing, stabilizing, binding agent	[92]
NP sols	Superhydrophobicity, oil-water separation, stain repellency, self-cleaning	Co, Bamboo, lignocellulosic	TiO <sub>2</sub> NP, SiO <sub>2</sub> NP, VTES	Ethanol	NaOH	-	[93]
<i>In situ</i> sol-gel	Antibacterial, abrasion resistance	Co	TEOS, zinc acetate	Ethanol, water	HF	-	[94]
<i>In situ</i> sol-gel	Photocatalytic activity, UV-protection	Co	Ti(OBu) <sub>4</sub>	<i>tert</i> -butanol	Acetic acid	-	[95]
<i>In situ</i> sol-gel	Thermal resistance	Co	GPTMS	Water	HCl	-	[96]
<i>In situ</i> sol-gel	Photocatalytic activity	PES, PLA	TIP	Ethanol	Acetic acid, HCl	-	[97, 98]
<i>In situ</i> sol-gel	Sweat pH sensing	Co	GPTMS	Water	BF <sub>3</sub> ·OEt <sub>2</sub>	Halochromic dye/litmus	[99]
<i>In situ</i> sol-gel	Flame retardancy	Wo	TEOS	Ethanol, water	HCl	H <sub>3</sub> BO <sub>3</sub> , Zn <sub>3</sub> B <sub>2</sub> O <sub>4</sub> , NH <sub>4</sub> FB <sub>2</sub> O <sub>4</sub> / flame retardants	[100]
<i>In situ</i> sol-gel	Hydrophobic, conductive	Co	MTMS	methanol	HCl	MWCNT	[101]

NP: nanoparticles, NH<sub>4</sub>OH: ammonium hydroxide, UPF: UV-protection factor, CV: viscose, PVP: polyvinylpyrrolidone, EG: ethylene glycol, UHMWPE: ultra-high-molecular-weight polyethylene, PVA: poly(vinyl alcohol), MPTMS: γ-methacryloxy propyl trimethoxy silane.  
 TIP: Titanium(IV) isopropoxide, CHPTMAC: 3- chloro-2-hydroxypropyl trimethylammonium chloride, AEAPTMS: N-[3-(trimethoxysilyl)propyl]-ethylene-diamina  
 PVS: polyvinylsilsequioxane, VTES: Vinyltriethoxysilane, PLA: poly(lactic acid), HF: Hydrofluoric acid, BF<sub>3</sub>·OEt<sub>2</sub>: Boron trifluoride diethyl etherate, MTMS: methyltrimethoxysilane, MWCNT: Multiwall carbon nanotube.

**Table 1.** Some researches about various functionalities on textile materials treated with *in situ* sol-gel method and sols with nanoparticles.

guanidine phosphate as a flame-retardant agent were prepared for using at pad-dry-cure process. On the other hand, the combination of the nanosols and various polymers such as polyacrylate, polyvinyl acetate (VA), and polyurethane dispersions and fluorocarbon polymer was applied to cotton fabric by knife-over-roll process. They found that the fabric coated with nanosols containing VA polymer relatively has good durable water-oil repellency and flame retardancy properties [103].

Chakrabarti and Banerjee prepared multifunctional cotton fabric that shows enhanced mechanical strength, water and stain repellence, antimicrobial properties, UV-blocking capacity, and self-cleaning characteristics by zinc oxide nanoparticles [104].

Ibrahim et al. enhanced both the functionality and pigment-printability of linen/cotton-blended fabric in a one-step process by the incorporation of inorganic nanomaterials such as silver (Ag-NPs), zinc oxide (ZnO-NPs), zirconium oxide (ZrO<sub>2</sub>-NPs), or titanium dioxide (TiO<sub>2</sub>-NPs) [105].

Mahlting et al. obtained photobactericidal and photochromic textile materials with UV-protective properties by sol-gel technology and stated that especially epoxysilane-modified sols led to promising results [106].

Pan et al. prepared superhydrophobic (water contact angle of 146.27°) and UV-blocking (UPF value of 164.06) cotton fabric by nano-alumina sol via sol-gel method [107].

Simonic et al. compared the sol-gel application procedures as one step or two steps in terms of multifunctional water-oil-repellent and antimicrobial properties of cotton and found that one-step procedure was more effective in the production of coatings with water- and oil-repellent properties, while two-step procedure provided higher antibacterial activity, as well as better washing fastness [108].

Mahlting and Fischer applied sol-gel coatings to viscose and polyamide textiles in order to give functional properties like water repellency and antimicrobial activity at the same time and found that textile comfort properties were also preserved [109].

Textor and Mahlting described a sol-gel-based surface treatment for the preparation of water-repellent antistatic textiles and applied it to two types of textiles (100% polyester fabric and 65% polyester and 35% cotton-blended fabric). They obtained sufficient water repellency and antistatic properties simultaneously and suggested that this concept can be adapted to the alternative multifunctional surface coatings other than combining water repellence with antistatic properties for textile materials [110].

El-Shafei et al. purposed to develop flame-retardant and antibacterial cotton fabric by treatment with BTCA solution containing sodium hypophosphite (SHP) as a catalyst and chitosan phosphate synthesized and then TiO<sub>2</sub> nanosols. Increase of TiO<sub>2</sub>, SHP and chitosan-phosphate concentration led to increase of LOI values of the fabric treated with them. The combination of BTCA and chitosan phosphate causes lower crease recovery angle than that of treatment with only BTCA. The antibacterial activity of the fabrics against *S. aureus*, *E. coli*, *Candida albicans* (fungus), and *Aspergillus flavus* (fungus) developed with the increase of TiO<sub>2</sub> and chitosan-phosphate concentration [111].

Rana et al. investigated the water repellency, UV protection, and antibacterial properties of cotton fabric treated with silica nanosols containing dimethyl dimethoxysilane (DMDMS) and TEOS as precursors and AgBr-TiO<sub>2</sub> nanoparticles, which were synthesized by using silver nitrate, potassium bromide, and titanium tetrachloride as precursors, by spray coating process. The treatment with silica nanosols with and without AgBr-TiO<sub>2</sub> particles gave rise to, respectively, 145.8°, 149.1° and 136°, 144.3° of contact angle values of the fabrics before and after washing process. Nevertheless, the fabrics treated with silica nanosols along with AgBr-TiO<sub>2</sub> particles have excellent UV protection with 41.9 of UPF value and good antibacterial activity with 99.88% bacterial reduction against *E. coli* [112].

Vasiljević et al. prepared six different solutions containing 1H,1H,2H,2H-perfluorooctyltriethoxysilane (SiF), 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (SiQ), and P,P-diphenyl- N-(3-(trimethoxysilyl)propyl) phosphinic amide (SiP) together with TEOS or organocyclotetrasiloxane 2,4,6,8-tetrakis (2-(diethoxy(methyl)silyl)ethyl)-2,4,6,8-tetramethyl-cyclotetrasiloxane. Optimization of water, oil repellency, flame-retardant, and antibacterial properties of the fabric treated were carried out. They revealed that the fabrics treated at optimized conditions have superhydrophobic properties with 161° of contact angle and 4° of sliding angle and good antibacterial properties with 100 and 81.6% bacterial reduction against, respectively, *S. aureus* and *E. coli* but not good oil repellency properties (3) [113].

Gu et al. proved the flame retardancy and water repellency properties of cotton fabric treated with phosphorus-doped silica hydrosol containing hexadecyltrimethoxysilane. They found that the LOI value, residue amount at 600°C, and contact angle of the fabrics increased from, respectively, 18.5, 13.3% and hydrophilic to 29.4, 29.7, and 134.6° when compared to that of untreated fabric. Their onset temperature and pHRR values decreased from, respectively, 320 and 235°C to 217 and 81.46°C [114].

El-Naggar et al. stated that the cotton fabrics treated with titania nanosols in situ synthesized and then urea nitrate solution by two-step process possessed excellent antibacterial properties with, respectively, 99.4 and 99.37% bacterial reduction against to *S. aureus* and *E. coli* and excellent UV-protection properties with 42.11% UPF value with good durability to washing [115].

Sivakumar et al. studied the antibacterial, UV-protection, soil release, and self-cleaning properties of the cotton/polyester fabric subjected to TiO<sub>2</sub> nanoparticles, which were synthesized using titanium isopropoxide as precursor and then modified with 3-aminopropyltriethoxysilane and silicon oil, by pad-dry-cure process. The maximum photocatalytic and antibacterial activities were obtained for the fabrics treated with modified TiO<sub>2</sub>, while the maximum UPF values were obtained for the fabric treated with unmodified TiO<sub>2</sub> [116].

Behzadnia et al. developed the photosonochemical synthesis of N-Ag/TiO<sub>2</sub> on wool fabric using titanium isopropoxide and silver nitrate as precursors and ammonia as nitrogen doping. The effect of pH, sonication, and precursor concentration on antibacterial and photocatalytic properties of wool fabrics treated were researched. Ag doping to N-TiO<sub>2</sub> nanocomposites on wool improved self-cleaning, photocatalytic, and antibacterial properties of N-doping TiO<sub>2</sub> nanocomposites on wool, while sono-treated samples containing N-Ag/TiO<sub>2</sub> possess higher self-cleaning and photocatalytic properties when compared to stirred fabrics containing N-Ag/TiO<sub>2</sub> [117].

### 3. Conclusion

Since 1960s, sol-gel-coating method on the substrates like metal, glass, and ceramic has been studied extensively. In the last decades, researches about sol-gel technology have focused on enabling functional properties to textile materials as an alternative to conventional textile finishing treatments. In textile finishing, flame retardancy, water or oil repellency, UV protection, antimicrobial activity, and antiwrinkle effect were already studied by sol-gel method. In addition, multifunctionality means that two or more of these properties are evaluated in combination and investigated in terms of washing durability, textile comfort, and physical properties. It is an important fact to ensure multifunctional properties in one step, and by nanotechnological sol-gel method, this problem can be achieved and moreover, eco-friendly treatments can be applied in textile finishing due to less harmful chemical use. Therefore, sol-gel technology is expected to be used in plenty of textile finishing processes and promising studies are the starting points for further developments in future.

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