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REVIEW PAPER: SOL-GEL AND HYBRID MATERIALS WITH SURFACE MODIFICATION FOR APPLICATIONS

Sol–gel technology for innovative fabric finishing—A Review

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Abstract Sol–gel technology continues to interest researchers from both industries and governmental institutions in many parts of the world decades after its discovery. It offers efficient and high-purity production of nanopowders, fibres, solid structures and thin-film coatings. Possible applications of sol–gel technology can be found in a wide range of sectors, such as pharmacy, medicine, construction, aerospace, transport, food industry, optics, agriculture, semiconductor devices, catalysis and biotechnology. Also in the textile sector, sol–gel technology is expected to lead the production of fabrics with completely novel properties or the combination of various functions in one fabric. The sol–gel reaction is easy to perform and does not require special conditions and high temperatures. The reaction consists of a series of simple hydrolysis and condensation reactions. This paper presents an overview of sol–gel technology and discusses the fabric functions that can be achieved by the technology.

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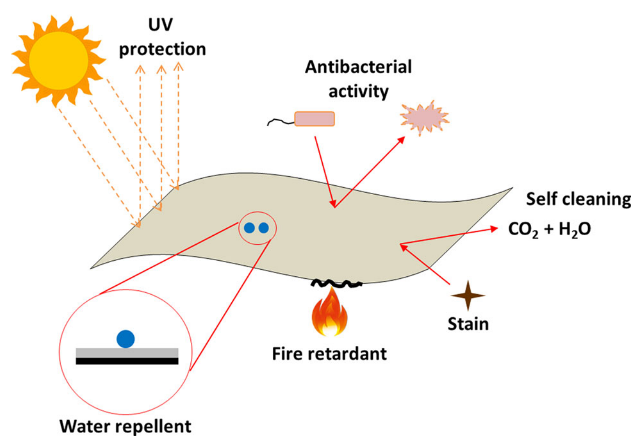
Graphical Abstract



Keywords Surface modification · Textiles · Fabrics · Finishing · Fabric functions

1 Introduction

Textile processing can be divided into three important stages: pretreatment, colouration and finishing [1]. Pretreatment process involves the preparation to dyeing, while colouration involves both dyeing and printing operations. Finishing is the final step in the fabric manufacturing process, to provide the properties and performances that customers will value and to give special functional properties. Most finishes are applied to fabrics such as wovens, knitwear or nonwovens. But there are also other finishing processes, such as yarn finishing, sewing yarn with silicones and garment finishing.

Textiles finishing can be subdivided into two distinctly different areas. The first one is chemical finishing (wet finishing), which involves the application of chemicals to textiles to achieve a desired result. Another one is mechanical finishing (dry finishing), which uses mainly physical means to change fabric properties and usually alters the fabric appearance as well. This finishing also encompasses thermal processes such as heat setting. The proper formulation of chemical finishes requires consideration of several important factors: the type of textile being treated (fibre and construction), the performance requirements of the finish (extent of effect and durability), the cost to benefit ratio, restrictions imposed on the process by availability of machinery, procedure requirements, environmental considerations, and compatibility of different formula components as well as the interaction of the finishing effects. To bring all these parameters to an acceptable compromise is not easy, even for a single purpose finish. But usually several types of finishes are combined for economic reasons mostly in one bath (only one application and drying process). This is often the hardest challenge of chemical finishing.

Chemical finishing has always been an important component of textile processing, but in recent years the trend to "high-tech" products has increased the interest and use of chemical finishes. As the use of high-performance textiles has grown, the need for chemical finishes to provide the fabric properties required in these special applications has grown accordingly [2]. In the last few years, sol–gel technology (chemical finishing technique for fabric) has real interesting application for the textile industry. This is mainly due to the versatility of their processing and the ease with which their properties may be tailored to fit a particular application. The unique and new properties of sol–gel materials have attracted not only scientists and researchers but also businesses, due to their huge economical potential.

The objective of this review is to discuss the sol–gel processing stages and its application in the chemical finishing of textiles. A list of sol–gel approaches that has been recently used in fabric finishing is provided in Table 1. Fabric functions discussed include ultraviolet (UV) protection, antibacterial, flame retardant, water repellent and self-cleaning.

2 Sol–gel technology

Sol–gel technology has become one of the most important emerging technologies in the fabric industry for many reasons such as more effective, low cost, less chemicals consumption, functionality and durability of important properties, and the most important is less environmental impacts [13]. Sol–gel technique is a common chemical approach to produce high-purity materials shaped such as powders, thin-film coatings, fibres, monoliths and self-supported bulk structures [14]. A sol–gel process, as the name implies, involves the evolution of inorganic networks through the formation of a colloidal suspension (sol) and gelation of a sol to form a network in a continuous liquid phase (gel). The starting compounds for preparation of a sol are precursors, which consist of a metal or metalloid element surrounded by various ligands. For this reason, precursors of various chemical structures can be used, whereas silicon alkoxides [$Si(OR)_4$] are the most common [15].

Metal alkoxides include reactive alkoxide groups (–OR), which react readily with water in the reaction of hydrolysis in the presence of a mineral acid or a base as a catalyst. The hydrolysis reaction replaces alkoxide groups with hydroxyl groups (–OH), which in the subsequent condensation reaction produce metaloxane bonds (M–O–M) (Fig. 1).

Table 1 List of sol–gel approaches that has been recently used in fabric finishing

Fabric	Sol–gel approach	Sol–gel material	References
Polyamide and polyester cotton	Super-hydrophobic water repellent	Hexadecyltrimethoxysilane and triethoxytridecafluorooctylsilane-doped Silica (SiO_2)	[3]
Cotton	Super-hydrophobic water repellent	Amino- and epoxy-functionalized silica	[4]
Polyester cotton	Antimicrobial	Octenidine-doped SiO_2	[5]
Aramid	Ultraviolet protection	Tetrabutyl titanate	[6]
Cotton	Antimicrobial	SiO_2	[7]
Cotton	Ultraviolet protection	Tetraethyl orthotitanate and tetraethyl orthosilicate	[8]
Cotton	Flame retardant	Phosphorus-doped SiO_2	[9]
Polyacrylonitrile	Flame retardant	Phosphorus-doped SiO_2	[10]
Viscose	Flame retardant	SiO_2	[11]
Cotton	Flame retardant	Phosphorus-doped SiO_2	[12]

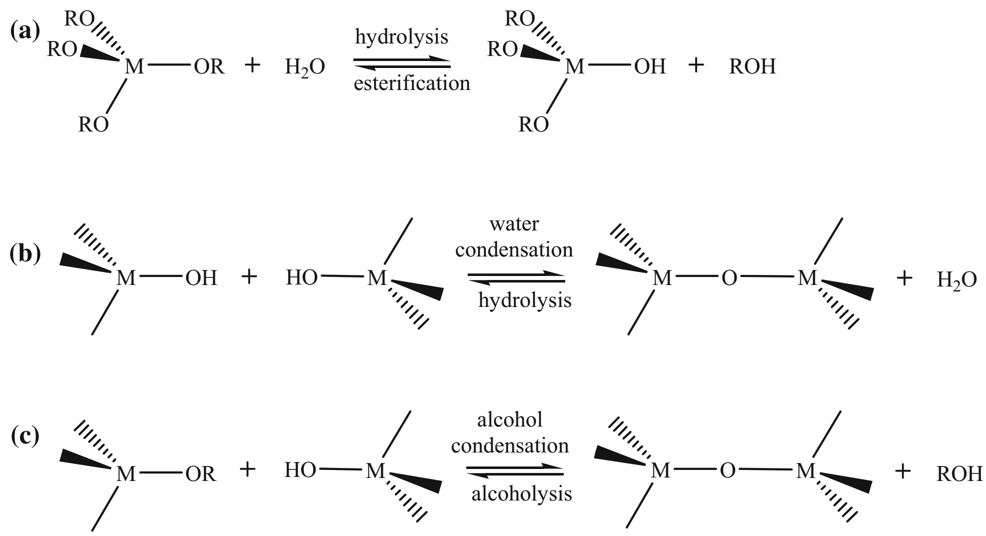


Fig. 1 Hydrolysis (**a**) and condensation (**b**, **c**) of metal alkoxides

This type of reaction can continue to build large metal containing polymer network with a three-dimensional structure by the process of polymerization (Fig. 2). When the polymers extend throughout the solution, they irreversibly form gel which upon drying affords amorphous xerogel with porous structure. The xerogel reforms into the crystallized polycondensate during heating at temperature of 150 °C. The characteristics and properties of a particular sol–gel network are related to a number of factors that affect the rate of hydrolysis and condensation reactions, such as pH, temperature and time of reaction, reagent concentrations, catalyst nature and concentration, water-to-metal precursor molar ratio, ageing temperature and time and drying.

Application of a sol–gel process in the chemical finishing of textiles includes a pad-dry-cure method, which consists of the impregnation of textile fibres by the sol following by the fibre drying and curing under the appropriate conditions. During drying and curing, the nanocomposite dense polymer film of thickness of some

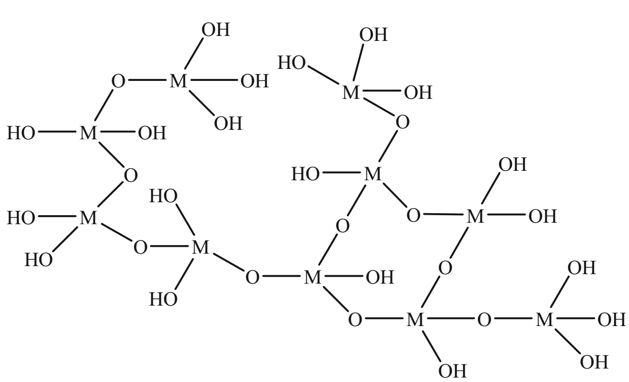


Fig. 2 Formation of polymer network by metal alkoxides with a three-dimensional structure

10 nm is formed on the fibres surface. The M–OH groups of precursors can also react with the fibre surface forming hydrogen (Fig. 3) as well as covalent bonds (Fig. 4). The latter, which is formed between the precursor's M–OH group and the hydroxyl group of the fibre in the reaction of condensation, strongly increases the adhesion of the polymer film to the textiles as well as the degree of polymer film orientation.

For chemical modification of textile fibres, they are a class of hybrid organic–inorganic materials which enable facile formation of network polymer films with high level of chemical functionality. The organic group is an integral part of the network architecture (Fig. 5) [16]. The organic–inorganic structure gives the polymer film dual properties, i.e. elasticity of polymer and hardness of ceramic. Due to the extremely thin polymer film, it does not cause any significant influence to the physical properties of the textiles such as tensile strength, softness and elasticity. Neither does it penetrate into the pores between the fibres, thus retaining the textiles' breathability. The treatment of textile fibres with hybrid organic–inorganic precursors opens numerous new possibilities for the improvement of their functional and protective properties [18–23], depending on the chemical structure of the organic group.

In recent years, the introduction of sol–gel nanocomposites in textile industry has attracted the attention of researchers. The ability of sol–gel process to form nanocomposites and structured materials from nanocoatings without disruption of their structure or functionality has provided exciting results in different areas of applications [24]. In fabric finishing, the application of nanocomposites materials is noted for its excellent hydrophobicity, oleophobicity, decreased inflammation, improved abrasion stability, electrical conductivity, UV protection, biocatalytic activity, anti-microbial activity and

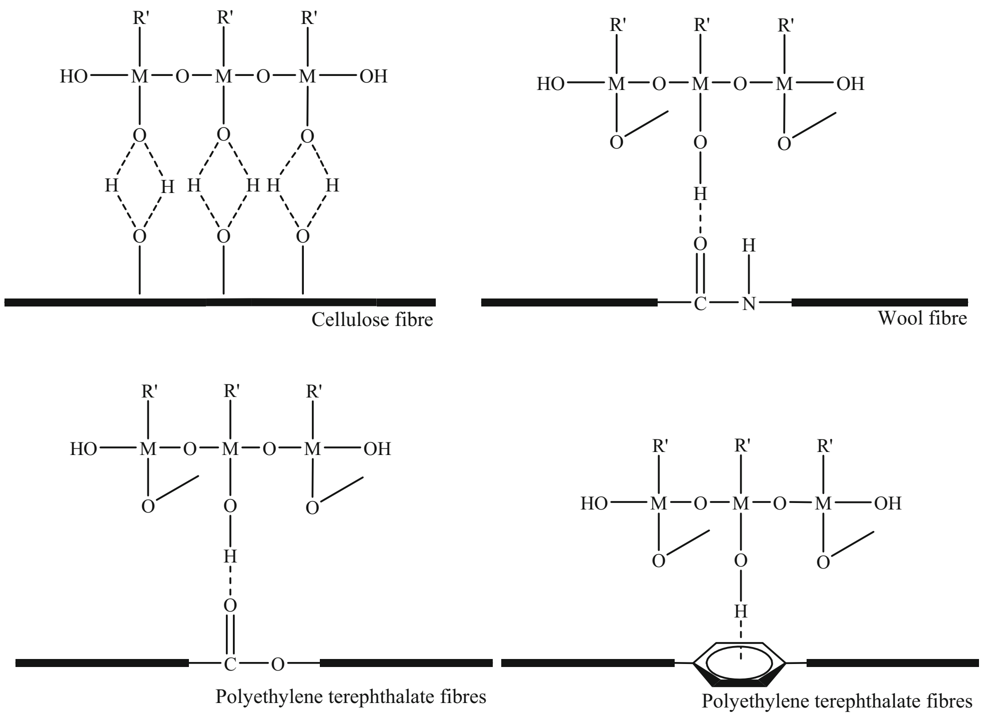


Fig. 3 Hydrogen binding of the precursor M–OH group to the fibre surface

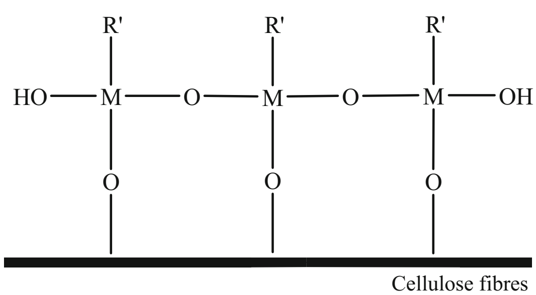


Fig. 4 Covalent binding of the precursor M–OH group to the hydroxyl group of the textile fibre in the reaction of condensation

controlled release of oils and flavours. Using a combination of different precursors with synergistic action in the mixture, multifunctional textile properties can be achieved.

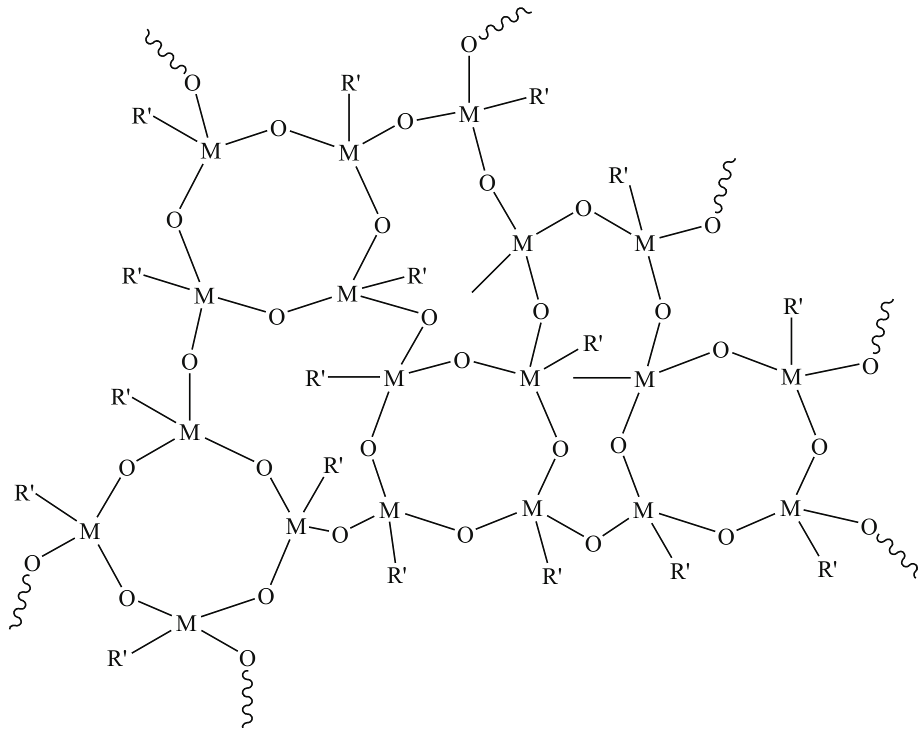
3 Fabric functions

Sol–gel technology has become one of the most important innovative finishing technologies in the fabric industries to impart the demanded functional properties and to cope with the need for revolutionary fabrics to face the great challenges in the global market without adversely affecting the environment. The imparted functional properties of fabrics are determined by type of substrate, finishing formulation, finishing technique, available equipments and performance requirements as well as economic and ecological aspects. In recent years, there has been increasing interest in improving fabric functionalities and performance properties by using sol–gel technology, including UV protection, antibacterial activity, flame retardant, water repellency, self-cleaning and dye ability.

3.1 UV protection

Over exposure to UV radiation can cause premature ageing and sunburn of the skin as well as degradation of fabric materials [25]. Thus, the enhancement in UV protection functionality is a great demand. Using protective clothes with high ultraviolet protection factor (UPF) values in addition to sunscreens is the main option of protection [26]. The requirements for a material to be effective as a UV protection finish include efficient absorption of UV radiation at 300–320 nm, quick transformation of the high UV energy into vibration energy, convenient to fabric fibres and lack of added colour for the treated fibres [2]. Figure 6 shows the mechanism of UV protection by nanosol-treated fabric. The nanosol works as UV blocking mainly through UV absorption, scattering and reflection mechanisms [27]. Previous study conducted by Alebeid and Zhao [28] has reported on the use of titania nanosol as modified fabric coating. They have prepared the nanosol with tetraisopropyl orthotitanate by sol–gel process and applied it on cationized cotton fabrics. They found out that the fabric has improved in terms of UPF values and also durability of

Fig. 5 A polymer network formed by suitable organofunctional metal alkoxides [17]



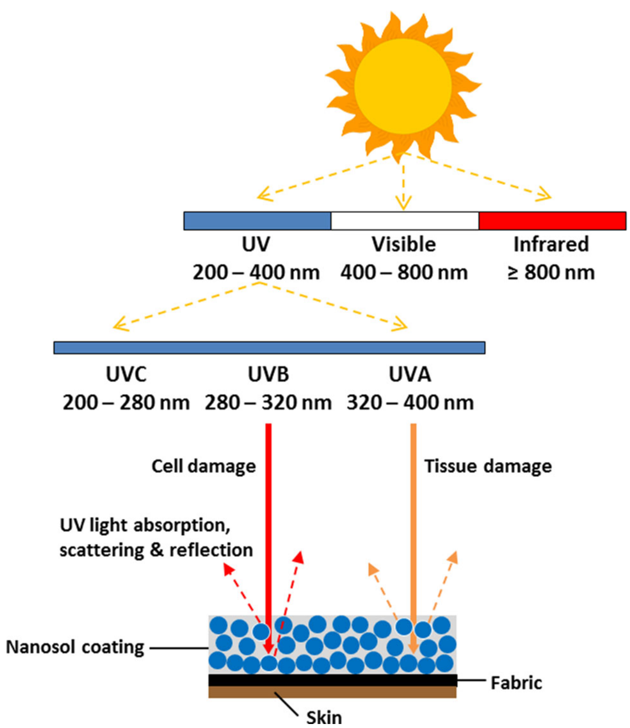


Fig. 6 Ultraviolet protection mechanism of nanosol-treated fabric

ultraviolet radiation protection. Sol–gel technology is very convenient to use in the preparation of UV protection materials of fabric since it allows the preparation and stabilization of a huge number of metal and metal oxide sols [29]. In addition, this technology has the ability to prepare thin layers and able to covers all fibres with high enough adhesion [30].

While Vihodceva and Kukle [31] have reported that zinc oxide (ZnO) nanosol prepared by using the sol–gel process can also be used to impart outstanding UV-blocking property to the finished cotton fabrics as it is low cost, white appearance, UV-blocking property and not harmful [13]. The sol–gel process was performed by the hydrolysis of tetraethoxysilane (TEOS) precursor in a mixture of water and alcohol in acidic conditions before mixed with zinc acetate. The ZnO nanosols obtained were then characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). They reported that the treated cotton fabric has excellent UV protection even though after 50 washing–drying cycles. This indicates that the sol–gel coating has very good adhesion with the fabric surface.

3.2 Antibacterial

The growth of microorganism on fabric has negative effects not only on the fabrics but also on the wearer, since it results in biodegradation of fabric materials along with their dissemination as a health risk [32]. An effective antibacterial finish should be quick acting, able to kill or stop the growth of microorganisms, durable to wash or dry cleaning, compatible with other finishing ingredients, environmental friendly, high quality but low cost, easy to apply and low toxicity [33]. As sol–gel technology has emerged as a promising tool to functionalize fabric surfaces, many previous studies have applied it for antibacterial treatment of fabric. Tarimala et al. [34] have used this technology to modify cotton fabric with dodecanethiol-capped silver nanoparticle-doped silica sol for antibacterial performance against *Escherichia coli* (*E. coli*). The sol was prepared with tetraethyl orthosilicate as the precursor and then added with the dodecanethiol-capped silver nanoparticles. The antibacterial performance was assessed for untreated cotton fabric, cotton fabric treated with the sol and cotton fabric treated with doped with dodecanethiol-capped silver nanoparticles. Their results showed the sol doped with dodecanethiol-capped silver nanoparticle exhibits inhibition rate of 40 % against the *E. coli* as compared to untreated cotton fabric [34]. In 2012, Prabhu and Poulose [35] have explained the antibacterial mechanism of silver nanoparticles. The silver nanoparticles are able to penetrate the bacterial cell wall and cause structural changes in the cell membrane and afterwards cell death (Fig. 7).

Sol–gel silica loaded with copper nanoparticles was used by Berendjchi et al. [36] to fabricate antibacterial surface on cotton fabric. The tetraethyl orthosilicate was hydrolysed and condensed in water to obtain the colloidal silica nanoparticles at room temperature. For antibacterial testing, two non-spore-forming bacteria were used, which are *E.coli* and *Staphylococcus aureus* (*S. aureus*). They reported that this technology has led to an efficient antibacterial activity of the modified fabric surface by reducing the percentage of bacteria growth more than 70 % for *E. coli* and 90 % for *S. aureus* bacteria [36]. The treated fabrics exhibited good antibacterial activity because of the settlements of copper nanoparticles on the silica nanoparticles as the antibacterial activity of metallic nanoparticles has a strong relationship with their sizes.

3.3 Flame retardant

Flame-retardant fabrics are very essentials to be used as safety and protective garments. Flame retardants make it more difficult for fabrics to ignite, make them burn slower and allow time to remove the clothing or put out the fires. Since a garment or a uniform constitutes the safety barrier between the wearer and the source of potential injury, its characteristics will determine the degree of injury suffered in case of an accident or an emergency operation. Conventionally, chemical additives (halogenated hydrocarbons) are being used to reduce the flammability of fabrics, but these have a number of side effects. If the fire occurs, the halogen will produce highly toxic and corrosive combustion gases and also may create several environmental problems. Recently, there is a growing interest in the use of sol–gel nanocoatings as flame retardants due to their inherent advantages features and performance superiority over traditional coatings [37]. It offers a simple and convenient route for the synthesis of advanced material system and for applying them as halogen free flame retardants.

Flame-retardant finishing of polyacrylonitrile (PAN) fabrics based on hybrid materials containing phosphorus compounds by sol–gel method was investigated by Yaman [10]. He prepared the silica sol by acidifying TEOS solution with 0.01 M HCl solution as a catalyst for acidic hydrolysis. Flame-retardant treatment was performed by adding phosphoric acid to the nanosols to make P-doped solutions and impregnating the PAN fabrics in the P-doped silica nanosols. The result was compared to an untreated control sample by measuring the flame spread times of the fabrics in the warp direction. For the untreated sample without P-doped silica nanosol, the fabrics burn with rapid flame and melt when burning which produce heavy dense black and toxic smoke, while PAN fabrics treated with 0.34 and 0.50 M of P-doped silica nanosol have not burned after exposing to flame for 15 s. The nonflammable properties of

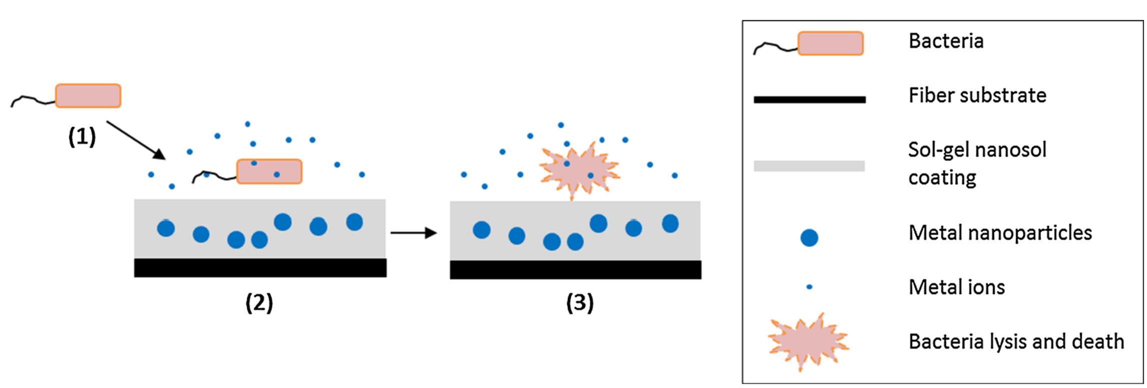


Fig. 7 Mechanism of antibacterial activity of nanosol-treated fabric: (*1*) bacterial invasion, (*2*) bacterial adhesion and metal ions binding, (*3*) bacterial lysis and death

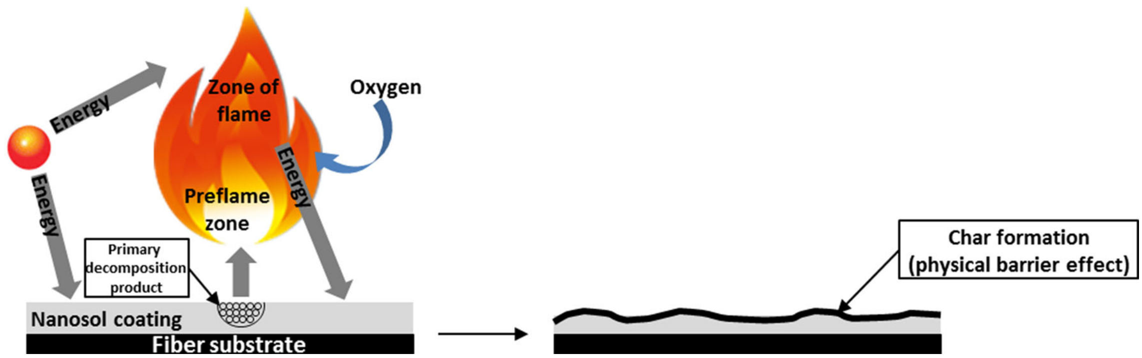


Fig. 8 Fire-retardant mechanism of nanosol-treated fabric

the treated fabrics were not completely lost after 10 times washing [10]. The action of phosphorus-containing flame retardants is known as "condensed phase" mechanism [38] where the phosphoric acid resulted from thermal decomposition will produce more residual char and less flammable by-products and stop the propagation of fibre (Fig. 8).

Zhang et al. [39] recently published a research paper focused on the functionalization of wool fabric with boron-doped silica sols as a flame-retardant finish. The sols were prepared by using sol–gel process involving the hydrolysis of tetraethyl silicate precursor. The flammability, combustibility, thermal stability and smoke suppressing property of the treated wool fabric were investigated. From the research conducted, they have found that the boron-doped silica sols have improved the flame retardant and thermal stability of the treated fabric while maintaining its tensile strength and air permeation [39].

3.4 Water repellent

Water-repellent finish should in the best case protect textile fabrics from wetting, without adversely affecting the air permeability of the finished fabrics, through reducing the free energy at the fibre surface. A surface tension of fabric surface is lower than that of the liquid is necessary to activate repellent finishes properties of the surfaces [29]. Low energy surfaces can be applied to fabrics through the chemical reactions of the repellent material with the fabric surface. Water repellency can be evaluated using the water drop test (Fig. 9), contact angles and spray test. Several techniques including the most effective sol–gel process have been used for the preparation of super-hydrophobic surfaces. Not only in textiles, but this technique was widely used in various fields in producing super-hydrophobic properties such as corrosion [40], optic [41], glass [42], paint [43] and automobiles [44].

Coating of fabric with silica nanosols containing perfluoroalkyl compounds has been used to achieve ultra-

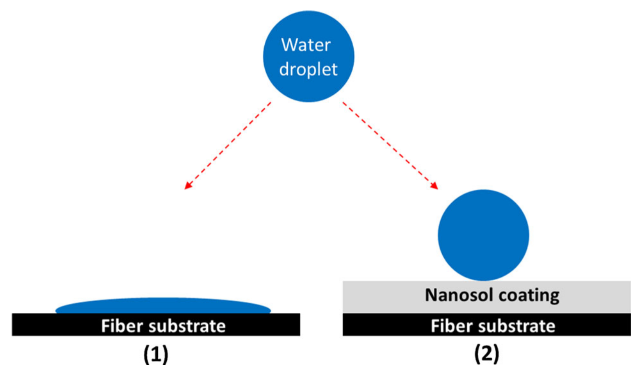


Fig. 9 Water-repellent mechanism of nanosol-treated fabric

hydrophobic, oleophobic as well as soil repellent functional properties [45–47]. Afterwards, non-fluorinated hydrophobic nanosols based on long-chain alkylsilane additives have been used to avoid the negative environmental impacts of fluorinated compounds as well as to attain coated fabrics with ultra-hydrophobic properties [3]. Moreover, many attempts have tried to increase the hydrophobic properties of fabric by using inorganic sols based on silica or titanium dioxide along with other hydrophobic additives [48, 49].

Instead of increasing hydrophobicity of fabric by coating process, conversion of naturally hydrophilic cellulose-based fibres such as cotton fabrics into hydrophobic matter would be a great alternative for the design and preparation of water- and oil-proof fabric with self-cleaning and anti-fouling properties [50]. Increasing demand towards renewable raw materials and more environmentally friendly and energy-efficient materials has made the modification of cellulose-based materials become more importance [51]. Cellulose substances can be used as template, scaffold or precursor [52]. These substances are suitable substrate for metal oxide film deposited by using sol–gel process with increasing hydrophobicity and oleophobicity characteristics [53, 54]. Jin et al. [50] have developed a cellulose-based material from commercial laboratory filter paper as a substrate for deposition of ultra-

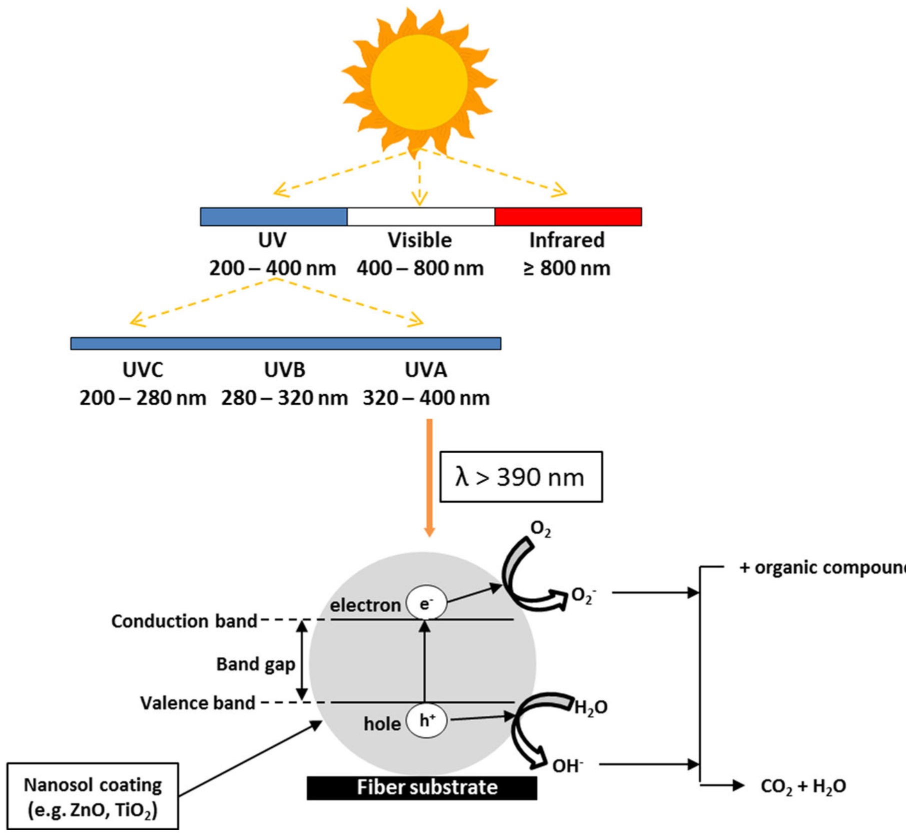


Fig. 10 Self-cleaning mechanism of nanosol-treated fabric

thin titania film via sol–gel process. They found that the surface of the filter paper has high hydrophobicity and oleophobicity characteristics that able to inhibit the adhesion of bacteria [51]. These findings promise an impressive future potential and wide range of applications of modified cellulose in textile industries.

3.5 Self-cleaning

Self-cleaning is referred to the photocatalytic activity, which uses light (photo) to activate a substance that modifies the rate of a chemical reaction without being included itself (catalysis) [54]. Self-cleaning mechanism is illustrated in Fig. 10. In the presence of light and moisture in the air, active oxygen is formed on the surface of nanosol-coated fabric. When organic compounds come into contact with the active oxygen, they are oxidized and broken down into harmless carbon dioxide and water vapour [55]. Photocatalytic self-cleaning materials include SnO_2, WO_3, CdS, ZnO and TiO_2, and among these materials, TiO_2 is most preferred because of its non-toxicity, chemical stability, low cost and durability against corrosion [56].

Previous studies [57–60] have reported on various synthesis methods of TiO_2, and among all the methods, sol–gel technique is the most used as it provides a simple and adjustable synthesis route at a relatively low temperature [61]. Gupta et al. and Elsayed et al. [59, 62] have performed the sol–gel synthesis of TiO_2 by the hydrolysis of a titanium precursor, namely titanium tetraisopropoxide in an acidic aqueous solution. Meanwhile, Kumar [60] has reported the use of titanium tetrachloride as the sol–gel precursor and hydrolysed by adding 1.0 M ammonia. In addition, Ortelli et al. [63] have come out with three different purification treatments of the commercial TiO_2 nanosol in order to purify and neutralize the nanosol in industrial scale. Based on their photocatalytic experiments, they have concluded that purification treatment involving the use of anion exchange resin is the most effective treatment as compared to ultra-filtration and neutralization treatments. Their findings are very significant to overcome the industrial scalability of self-cleaning textile production limitation [63]. From the previous studies mentioned, sol–gel technique is a promising tool to synthesis self-cleaning materials at low temperature since textile substrates have poor thermal stability [59].

The increasing interest on nanosol coatings for self-cleaning photocatalytic textile production has inspired a discovery of a new method known as dip-pad-dry-cure [64]. This method was introduced by immobilizing the nano-TiO_2 onto cellulose substrates in order to exploit the photocatalytic reactivity of the nano-TiO_2. The new method has produced stable and reproducible multihelical titania nanotubes, which provide a low cost and efficient route to produce unique structural features of ceramic nanomaterials in future.

4 Multifunctional

In previously reported studies, sol–gel technique can be used to impart multifunctional properties onto treated fabric, mixing different organic molecules functionalizing or choosing appropriately the inorganic precursors. As an attractive multifunctional material, nanosized TiO_2 has attracted attention [65]. Multifunctional properties of TiO_2 nanoparticles have been reported by Kathirvelu et al. [54], and the study was conducted on cotton and polyester/cotton blended fabric. The nanoparticles were synthesized using sol–gel technique where titanium tetrachloride was used as a precursor and nitric acid as a catalyst. The study revealed that both treated cotton and blended fabrics had various functionalities including antimicrobial activity, self-cleaning and UV protection properties [54].

Dhineshbabu et al. [66] have coated sol–gel anatase TiO_2 nanoparticles and also TiO_2 nanoparticles bind with silica (SiO_2) on cotton fabrics by pad-dry-cure method. The study concluded that the hybrid TiO_2/SiO_2 nanoparticle-coated fabrics show improved multifunctional properties such as antibacterial activity, UV blocking and fire resistance which suitable for use in biomedical, industrial and defence fields [66]. Recently, another study was conducted to investigate the multifunctional properties of TiO_2 nanoparticles, which were synthesized using titanium tetraisopropoxide as the precursor in the presence of polycarboxylic acid and sodium hypophosphite as catalyst [67]. They found that the treated cotton fabric reveals excellent flame retardant and antibacterial properties.

5 Conclusion

Sol–gel science and technology promises the possibility to modify the properties of materials and to combine different functionalities in a single material. One of the most significant benefits of sol–gel technology is the use of low temperature conditions and insensitivity to the atmosphere. These features will allow its use with various materials including the textile substrates which cannot tolerate high temperatures. The combination of innovative sol–gel materials and specific functionalization enables new perspectives for innovative fabric finishing with UV protection, antibacterial, flame retardant, water repellent, self-cleaning properties or the combination of the properties. It is likely that technical reports and peer-reviewed publications will come out of these works in the near future.

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