

Quaternary Ammonium-based Composite Particles for Antibacterial Finishing of Cotton-based Textiles

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The antibacterial finishing of cotton-based fabrics has been achieved from quaternary ammonium-based composite particles. This functionalization is based on the simple dilution of a quaternary ammonium cation (QAC) hybrid alkoxide within a sol–gel derived crystalline suspension (CS) of TiO₂ in liquid solution. This protocol yields the preparation of QAC–TiO₂ (QT) composite sols by using a same CS over a long period of time, and enables an easy regeneration of derived QT sols after quite long aging periods. Composite sols can then be impregnated on various kinds of substrates, including textile fabrics. Fourier transform infrared spectroscopy studies, as well as optical and scanning electron microscopy observations, have been used to investigate chemical and morphological features arising from QT particles. Antibacterial tests have then been performed on so-finished textiles and are discussed in relation to chemical and morphological features. It is shown that this sol–gel route flexibly yields a similarly strong antibacterial activity on cotton-based fabrics against both Gram-negative and Gram-positive bacteria, *i.e.* tested fabrics exhibit an antibacterial activity (according to the ISO 20743-2005 standard) ranging between 7.5 and 7.9 against both kinds of bacteria tested here. Finishing treatments also allow preserving the hand feeling and visual aspect of the textiles and promote a rather good attachment of impregnated particles on the surface of textile fibers.

KEY WORDS: Quaternary ammonium; TiO₂; Antibacterial efficiency; Textile; Sol–gel

1. Introduction

Textiles with enhanced resistance against micro-organisms (antibacterial, anti-microbial, antifungal textiles, or else) are becoming an increasingly desirable aim of textile manufacturers^[1–4]. Finishing treatments are particularly used to prevent three undesirable effects in textiles. The first includes the degradation phenomena like coloring, staining and deterioration of fibers^[5–7]. Because of their dye degradation potential, even some fungus can be used for removing dye from textile effluent^[8]. The second one produces unpleasant odor^[9–11] and the third effect is the increase of potential health risks^[12–14]. For instance, most finished textile materials, currently introduced in hospitals and food industries, are designed to protect wearers against the spread of nosocomial infections, inhibit the multiplication of pathogen micro-organisms, and remove the infection

sources. In general, antibacterial properties can be imparted to the textile materials by chemically or physically incorporating functional agents onto fibers of fabrics. Among numerous available varieties of antibacterial agents, species containing a quaternary ammonium cation (QAC) have been widely used for decades to disinfect environmental surfaces, for instance in clinical and industrial settings. This particular interest arises from inherent advantages, including a broad activity spectrum against Gram-negative and Gram-positive bacteria, yeasts, or moulds, as well as effectiveness over a wide pH range^[15–19]. Derived compounds are used in a variety of commercial applications ranging from cosmetic preservatives to hospital disinfectants and sanitizers. QAC-derived compounds, designed to impart antibacterial functionalization on textile fabrics, have already been submitted to many evaluations and commercialized^[1,4,20]. Besides, they still make the object of intensive investigations. In the recent literature, many works have, for instance, been proposed to improve the antibacterial finishing procedure and the performances of finished textiles. Deposition of QAC-derived compounds can be achieved through physical techniques on various kinds of supports^[21], but the antibacterial finishing of textiles is generally realized through the preliminary chemical preparation of QAC-based liquid solutions, which can in turn be

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impregnated by using reduced cost techniques such as pad-, spray-, foam-deposition methods, or else^[22]. Such liquid solutions can, for instance, be prepared through all-organic chemistry methods^[23–25].

Another convenient way to impart particular functionalities on textiles from liquid solutions relies on the implementation of sol–gel methods. In these methods, metal (M) alkoxides ($M^N(OR)_n$) react in liquid solutions through the hydrolysis and polycondensation of alkoxy (OR) groups (where R is an alkyl radical). These reactions yield the formation of an inorganic polymeric network constituted by more or less developed metal–oxygen chains. Some authors have taken advantage of such methods to physically incorporate QAC-based all-organic species within an inorganic silica network deposited on the surface of textile fibers^[26–28]. These authors reported on good antibacterial performances of so-finished textile fabrics. Organic species can also be deposited through sol–gel routes by using a so-called hybrid approach. In this approach, one (or more) alkoxy groups of a silicon alkoxide precursor are partially substituted by one (or more) organic radicals non reactive to sol–gel reactions. In this approach, sol–gel reactions result in the formation of an inorganic silica network incorporating organic species covalently bonded (*i.e.* firmly fixed) to the inorganic network. Over the past decade, so-formed hybrid materials have extensively been studied to formulate coupling agent for fiber/polymer composites or to induce various functionalities on textile fabrics (see reviews Ref.^[29,30]). However, hybrid alkoxides containing long organic chains are generally poorly reactive to sol–gel reactions^[31,32]. It is due to the steric hindrance provoked by long organic chains that reduces the chemical interactions between reactive alkoxy groups. When derived sols are deposited on a substrate, their lack of reactivity promotes undesirable dewetting effects at the substrate surface, owing to an insufficiently developed inorganic network that cannot efficiently counteract capillary forces occurring during the sol–gel transformation. Specific and rather complex procedures and protocols have thus to be set up to activate the sol–gel reactions in sols based on such hybrid alkoxides. On the one hand, sol–gel approaches by using a QAC hybrid alkoxide (QACHA) have already been investigated for different (non antibacterial) applications^[33–35]. On the other hand, some commercialized products intended to impart antibacterial properties on various supports, including textiles, are based on a QACHA diluted in liquid solution^[4,20], and some very recent works have also reported on the antibacterial functionalization of textiles from QACHA precursors^[4,20]. However, we surprisingly note that the literature devoted to such functionalization by using QACHAs is quite poor.

In this article, we present a hybrid sol–gel approach yielding QACHA–TiO₂ (QT) composite particles intended to impart an efficient antibacterial functionalization on textile fabrics. For industrial applications, it is important (i) to propose simplified elaboration protocols of liquid solutions, which reduce the fabrication costs, and (ii) to optimize the in-time stability of resulting sols, which allows the long term reproducibility of functionalized products derived from a same sol and reduces the need to frequently repeat sol elaboration operations. In a previous work, we proposed a hybrid sol–gel approach meeting such criteria, which was designed to impart a hydrophobic functionalization on naturally hydrophilic cotton-based fabrics^[36]. This approach was firstly based on the sol–gel preparation of a liquid suspension containing TiO₂ nanoparticles (NPs). As discussed in

our previous article^[36], it might be considered that this preliminary stage was quite complex, but the derived sol–gel procedure relied on two essential assesses: (i) the resulting TiO₂ liquid suspension exhibited an enhanced stability over time and could be used in reproducible conditions for weeks or months, and (ii) a hybrid alkoxide constituted of hydrophobic long alkyl chains (C16) could easily be grafted on TiO₂ NPs through a simple dilution of this alkoxide within TiO₂ suspensions, just before textile impregnation and without the need of any complex sol–gel formulation or protocol. This latter feature relied on the high surface reactivity of TiO₂ NPs, *i.e.* their ability to graft silicon alkoxides through Ti–O–Si surface bonds formed by hetero-condensation reactions, which led to C16–TiO₂ composite particles. In the present article, we study how this grafting method can be extrapolated to the antibacterial functionalization of textile fabrics by using a QACHA precursor. We firstly show how the formulation of so-formed QACHA–TiO₂ (QT) composite sols influences the chemical composition of derived species deposited on the surface of silicon wafers, chosen as model supports, together with the morphology of so-obtained coatings. Results are then extrapolated to the antibacterial functionalization of cotton-based textiles.

2. Experimental

2.1. TiO₂ sol preparation

The functionalization of textile fabrics with QACHA species is based on the preliminary preparation of a TiO₂ crystalline suspension through a two-step sol–gel route. A polymeric mother solution (MS) was first prepared by mixing tetraisopropyl orthotitanate (TIPT from Fluka) with deionized water, hydrochloric acid, and absolute ethanol as a solvent, according to our previously published procedure^[37]. TIPT concentration in the solution was 0.4 mol/L, and the TIPT/H₂O/HCl molar composition was 1/0.82/0.13. The solution was aged at room temperature for two days before use. Then, a crystalline suspension (CS) of TiO₂ nano-crystallites in absolute ethanol was prepared from the MS, by using a procedure that has also been developed in our group and previously detailed^[38]. Briefly, the MS was first diluted in an excess of deionized water (H₂O/TIPT molar ratio of 90) and autoclaved at 130 °C for 6 h. Autoclaving yielded the crystallization of TiO₂ NPs diluted in the aqueous medium. An exchange procedure was then performed in order to remove water from the sol and form a CS in absolute ethanol. The final TiO₂ concentration in ethanol was 0.24 mol/L. The CS was composed of TiO₂ NPs with a diameter of about 5–6 nm, crystallized in the anatase phase, and agglomerated in small polycrystalline aggregates of around 50–100 nm in size^[39]. Previous works have shown that CS preparation conditions give rise to very stable sols, *i.e.* no further particle aggregation takes place over a prolonged aging at room temperature^[38]. This stabilization is due to peptization effects induced by the acidic conditions, *i.e.* electrostatic repulsions between initially formed, positively charged, TiO₂ aggregates. Consequently, these suspensions can be stored for several weeks or months before use. Such suspensions have extensively been applied in our group to deposit and study photocatalytic TiO₂ films of high homogeneity and optical quality, which can in particular be deposited on thermally sensitive substrates through low temperature protocols^[38,39]. In the present work, we study how these suspensions allow envisaging the antibacterial functionalization of

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