



A sol–gel based surface treatment for preparation of water repellent antistatic textiles

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ABSTRACT

In this paper a surface treatment is described for preparation of hydrophobic sol–gel coatings that simultaneously offer antistatic properties for an appropriate finishing of textiles and refinement of polymer foils. Sol–gel based formulations are modified with both hydrophilic and hydrophobic components simultaneously. Hydrophobic components are, e.g., alkoxysilanes modified with alkyl chains while the hydrophilic ones are amino-functionalized alkoxysilanes. The basic idea is that due to an enrichment of hydrophobic groups at the solid/air interface the surface of the as prepared coatings will be hydrophobic while the deeper region will be more hydrophilic. Textiles finished with these coatings exhibit sufficient water repellence and simultaneously absorb sufficient amounts of humidity in the deeper areas of the coating guaranteeing antistatic properties. This concept offers interesting approaches for the preparation of multifunctional surface coatings not only focussing on combining water repellence with antistatic properties for textile materials.

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1. Introduction

The surfaces of materials exhibiting a low surface conductivity tend to charging under certain conditions. The phenomenon of charging occurs, e.g., when the humidity in an environment is low, this is well known in winter times when the compartment air is extremely dry. In an industrial environment the charging can be a serious risk. Spark formation might cause the ignition of flammable dusts, gases or liquids. The charging of synthetic polymer surfaces can exceed 35,000 V [1] discharging might therefore also be a problem for microelectronic devices that might be destroyed.

As well-known charging can be developed if contacting surfaces are separated. Parameters determining the charge formation are the specific materials separated (conductivity), the size and topography of the surfaces, the separation velocity, friction as well as the present atmospheric moisture. An accepted assumption is that a textile material exhibiting a surface resistance of less than $5 \times 10^9 \Omega$ will not build up enough charging for a spark formation [2]. Intrinsically conductive materials as, e.g., metals or conducting polymers will therefore not yield a relevant charging. Hydrophilic materials such as, e.g., cotton, that are not intrinsically conducting will also not cause problems at average climate conditions. Certain amounts of water stored by the materials guarantee a sufficient conductivity to prevent a high charging of several textile materials,

so the surface resistance is correlated to the humidity up-take of textile fiber. The very hydrophilic materials as cotton or viscose store high amounts of water (up to 12 wt.%) which results in a low surface resistance [2]. In comparison the more hydrophobic synthetic polymers take up less water resulting in a higher surface resistance. Polyethylene terephthalate fibers, e.g., only store about 0.2 wt.% of water.

Different approaches are known to prevent electrostatic charging of different materials by generating intrinsically conducting surfaces or bulk materials. Such as incorporation of comparably high amounts of, e.g., carbon black into a polymer, deposition of conductive layers on the materials surface by printing, coating, chemical or physical vapour deposition, co-weaving of metal threads or the use of carbon nanotube materials [3–5]. Another approach widely used in textile industries is the finishing with hydrophilic materials. The textile materials are impregnated with finishing agents containing anionic surfactants (e.g., alkyl sulfonates, alkylaryl sulfonates), non-ionic or cationic surfactants (quaternary ammonia compounds). Once applied to the textile these compounds absorb certain amounts of water resulting in a promising antistatic effect [6]. Most of the mentioned techniques are disadvantageous for different reasons; the PVD as well as the CVD processes are comparably costly, the incorporation of carbon black changes the material's properties. The durability of the mentioned impregnation with surfactants is mostly not sufficient. Simultaneously the increase of surface energy necessary to increase the amount of absorbed water is accompanied by an increased soiling or a fall off in cleanability. Conventional anti-

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soiling treatments employing, e.g., fluorocarbon polymers will increase the problem of electrostatic charging since these are insulators as the most synthetic polymers and will furthermore lower the amount of absorbed water due to its low surface energy.

Therefore, the scope of the presented work here was to combine both a low surface energy (aiming on water-repellency and anti-soiling properties) with a satisfying surface resistance. The sol-gel technique has been reported as a promising tool for preparation of water repellent coatings [7–12] especially versatile for application on textile [13,14] or wood [15]. Employing organically modified alkoxy silanes modified with long-chained aliphatic or highly fluorinated groups sol-gel route offers far reaching possibilities to prepare water as well as oil repellent textiles.

Besides equipping textiles with hydrophobic properties sol-gel technique can also be used to apply antistatic coatings. Several different methods are reported as the embedding of conductive organic polymers into silica sols, application of alumina containing sols, modifications with hydrophilic polymers, addition of non-ionic surfactant into alumina sols, there are also reports of ZnO containing recipes or the modification of silica sols with aminosilanes [16–25]. Different aminoalkyltrialkoxysilane containing sols are also described for preparation of proton conductive coatings on membranes [26,27]. Another interesting approach to realize antistatic sol-gel coatings is based on the cohydrolysis of precursors such as tetrathiafulvalene (TTF) with silicon alkoxides [28,29]. The use of TTF containing sol-gel coatings for modification of polypropylene or polyester fibers has been recently reported [30].

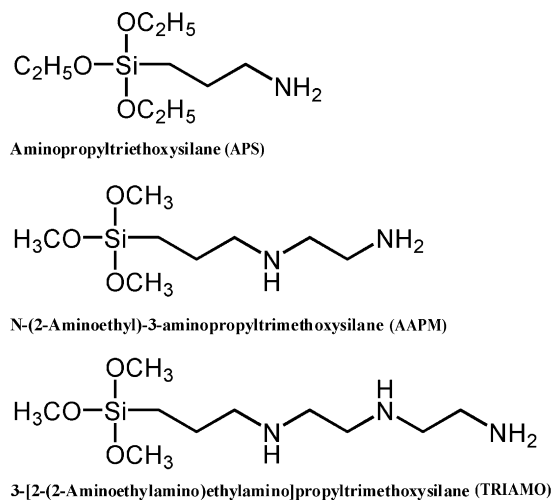
It has been reported that hydrophobic sol-gel based coatings can be prepared by combining, e.g., simple TEOS-based sols with minor amounts of hydrophobic additives as mentioned above. Amounts of 1–2 vol.% are reported to be sufficient to increase the repellence dramatically. An explanation for this is given, e.g., by Fabbri et al. [31]. Driven by the differences in the surface tension of the components the hydrophobic components enrich at the solid/air interface during the coating and drying process yielding low surface energy while the bulk of the coating will be less hydrophobic [31]. Analogously a hydrophilic additive is expected to yield an inverse effect, namely the hydrophilic components will enrich in the bulk of the coating. In that case, a combination of both hydrophilic and hydrophobic additives could yield a low energy surface and a hydrophilic bulk. If the hydrophilic bulk is furthermore able to store/absorb sufficient amounts of water/water vapour the resulting coating would guarantee a sufficient conductivity leading to sufficient antistatic properties.

2 Experimental

2.1 Sample preparation

For all solutions ethanol (96 vol.%) is used as solvent. Hydrophobic modified silica sols were prepared by acidic hydrolysis of tetraethoxysilane (TEOS) under addition of different alkyltrialkoxysilanes, as methyltrimethoxysilane (C1), butyltrimethoxysilane (C4), octyltrimethoxysilane (C8), dodecyltrimethoxysilane (C12), hexadecyltrimethoxysilane (C16) and octadecyltrimethoxysilane (C18). Triethoxytridecafluorooctylsilane (*F-silane*) (from Evonik) is alternatively used as hydrophobic and fluorinated compound. For preparation 20 ml of TEOS is stirred into 165 ml ethanol. The hydrophobic modification is carried out by replacing TEOS by one of the mentioned alkyltrialkoxysilane in a volume ratio ranging from 10 to 50 vol.%. After stirring this mixture for 5 min the hydrolysis is started by adding 36 ml acetic acid (50%) and the solution is stirred for 24 h.

To prepare hydrophobic silica sols additionally modified with amino functional groups different aminoalkyltrialkoxysilanes are



Scheme 1. Aminosilane additives used.

used, as aminopropyltrimethoxysilane (APS), N-(2-aminoethyl)-3-aminopropyltrimethoxysilane (AAPM) and 3-[2-(2-aminoethylamino)ethylamino]propyltrimethoxysilane (TRIAMO) (Scheme 1). These amino compounds are added under stirring to the mixtures of TEOS, alkyltrialkoxysilane and ethanol. After stirring these mixtures for 5 min, the hydrolysis is started by adding 36 ml acetic acid (50%) and the solution is stirred for another 24 h. The share of the aminosilane is adjusted to concentrations up to 17.8 vol.%.

The gained solutions are applied to two types of textiles, either a 100% polyester fabric (polyethylene terephthalate, PET) or a PET/cotton blended fabric (65% PET and 35% cotton). Both fabrics are plane woven, the weight of the PET fabric is 103 g/m² and of the PET/cotton fabric is 215 g/m². The application on textiles is performed by dip-coating, afterwards the samples are allowed to dry at room temperature before thermally treated at 120 °C for 30 min. Coatings on glass are produced analogously.

2.2 Analytic methods

The hydrophobic or water repellent properties of coated textiles are determined by the DuPont test (AATCC-118-1972-Test). The DuPont test evaluates the repellence of textile material for water and liquids with lower polarity. Evaluation is carried out employing eight different mixtures of water with iso-propanol [14]. At least three drops of each mixture are dripped onto the sample fixed in an embroidery hoop. Afterwards it is noted whether the different mixtures wet the fabric within 60 s. The mixture with the highest proportion of iso-propanol not wetting the textile within 1 min determines the result of the DuPont test; a grading of 8 is highest water repellence. In addition to the DuPont test, the TEGEWA drop test [32] was used to evaluate the water repellent properties of the coated textiles. In this test drops of water are applied to the textile from a certain height and the time until the drop is soaked up completely is measured. If the drop of water is not soaked up after 60 min the measurement is stopped and the TEGEWA value is set to 60 min. These measurements are performed at six different sample areas (front and back) and the average of these six measurements is given.

The antistatic properties of coated textiles are evaluated by determining the surface resistance according to DIN 54345. An annular electrode is employed to measure the flat material's (as, e.g., textiles) conductivity (the setup has been depicted, e.g., in [14]). The samples have been climatized for at least 24 h to guarantee reproducible results (*T* = 20 °C, rel. humidity = 65%), the measurements are carried out at the same climate conditions.

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