

# Synthesis and Fabrication of Silica Based Superhydrophobic Surfaces

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## Abstract

Superhydrophobic surfaces with very high water contact angle  $>150^\circ$  and very low roll-off angle have received more attentions due to their huge potential in environment, energy and biomedical applications. It is now well known that superhydrophobicity arises from a combination of low surface free energy and surface roughness. Superhydrophobic coatings were fabricated by spraying the silica hybrid emulsion on clean glass substrates. Then, the effects of the emulsion dosage and the mole ratio of silica on the coating performance were analyzed. Finally, the formation mechanism of the hybrid coating was discussed, and the self-cleaning properties of the superhydrophobic and hydrophobic coating tilting at the same angle were also compared. The results show that the hydrophobic silica nanoparticles can form rough micro/nanostructure on the coating surface, since they act as a solid plasticizer capable of enhancing the surface properties. This work is divided in the following sections

1. Introduction
2. Mechanism of the sol-gel process
3. Literature review
4. Conclusion

**Keywords:** Surface coating; Silica coating; Hydrophobicity

## Introduction

Learning from nature is an important source of new techniques and advanced materials. A typical example of this is the creation of superhydrophobic surfaces by bionic copying of the microstructure of lotus leaves [1-6]. On these superhydrophobic surfaces, water droplets

remain nearly spherical and easily roll off, removing dirt in their path [7] without wetting the surface [8,9]. The superhydrophobic behavior of lotus leaves, known as the lotus leaf effect or self-cleaning effect, resulted from the hierarchical rough structure, as well as the wax layer present on the leaf surface [10,11]. It has been well established that the wettability of a solid surface is

governed by both the surface chemical composition and the surface geometrical microstructures [12-16]. Superhydrophobic surfaces are defined as surfaces with water contact angle greater than  $150^\circ$  [17,18] and sliding angle below  $10^\circ$  [19,20]. Superhydrophobicity arises from a combination of low surface free energy and surface roughness [21]. There are two general methods for fabrication of artificial superhydrophobic surfaces: roughening of hydrophobic surfaces [22-24] and decrease the surface energy of a rough surface by chemically bonding low surface-energy species to the surface (i.e. modifying the rough surfaces with low surface free energy materials) [24-31]. In other words, to fabricate a surface with a water contact angle larger than  $150^\circ$ , two key factors must be considered; 1) When the surface energy is lowered, the hydrophobicity is enhanced. However, even the material with the lowest surface energy gives a water contact angle of only around  $120^\circ$  [32]; 2) very large contact angle and a superhydrophobic behavior can be determined only if low surface energy and elevated roughness are simultaneously present. The superhydrophobic behavior of a solid surface depends on the synergistic contribution of surface chemical composition (i.e. low energy materials) and unique morphologies (e.g. micro-nano hierarchical structure, a porous nanostructure) [33,34]. Superhydrophobic surfaces with high water contact angle acquire increased attentions due to their huge potential application in environment, energy and biomedical fields [13,35,36], oil-water separation [37], self-cleaning [38] or water directional flowing [39], anticorrosion, anti-fouling, anti-frost/anti-icing and antimicrobial [28,33,40,41], electronic and optical devices[42], photonic materials[43], oil water separation [44,45], drag reduction [46-48], anti-icing [49] and templates for fabricating bio and chemical sensors, and microfluidics [24,50,51]. Moreover, combining the synthesis of a superhydrophobic structure with materials that are responsive to different external stimuli such as mechanical force, temperature, or magnetic and electric fields, is currently of major interest in the materials science research community [52-54]. Superhydrophobic surfaces without high mechanical stability can hardly play their roles in the practical application. In fact, a large majority of superhydrophobic surfaces have very limited mechanical stability, they are prone to be damaged even after finger wipe [55]. Recently, a great deal of effort has been made to fabricate coatings, possessing the properties of superhydrophobicity and durability. Basically, hierarchical surface structure and low-surface-energy materials are both essential for the preparation of superhydrophobic surfaces [56]. The hierarchical dual-size surface roughness can allow air

chamber to form between the water droplet and surface texture, thus facilitating water droplet to roll off easily [5,57-60]. Hierarchical structure synthesized by template based techniques [2,12,61,62], chemical etching [63], deposition [64], electrodeposition[65], electrospinning [66], sol-gel method [67], and phase separation methods [68], electrochemical layer by layer deposition and colloidal assembly [69-71], plasma modification [72,73], chemical vapor deposition[74,75], calcinations[76], photolithography [77], UV-curable reaction [78], anodization methods [79], solution immersion [80], spray [81,82], and polymer imprinting [83] using polymers and metals. The most common method for preparing superhydrophobic surfaces is by spraying polymer solutions mixed with abundant inorganic particles to build rich multi-level structures which realize surface superhydrophobicity [40] through sol-gel method. Sol-gel methods preferred due to; 1) its mild operation situations without extra post-treatments such as high-energy etching or high-temperature calcination; 2) controlled surface morphology and enhanced coating properties [36]. Low-surface-energy materials comprise fluoride, hydrophobic polymers polyacrylate (PA) [84], poly(dimethylsiloxanes) (PDMS) [85], polyurethane (PU) [86], polypropylene (PP) [87] and polyaniline (PANI) [88]. Moreover, silicon, silica, polyelectrolyte/silica, polyelectrolyte/metal, raspberry-like particles and compounds with alkyl groups are used as low surface energy materials to fabricate superhydrophobic surface [8, 6]. Silica is one of the most popular used inorganic nanomaterials in the preparation of inorganic/organic composite materials [16,89-93] through typical emulsion polymerization [94] and produced by numerous companies. Conventional silica particles possess plentiful hydroxyl groups on their surfaces. This often requires the surface modification of  $\text{SiO}_2$  to enhance the compatibility with organic matters, which is limited by cumbersome procedure and inefficient surface grafting [16]. Mass-produced silica nanoparticles are available in the nanoscale and microscale at a very low cost. Silica nanoparticles are commonly used in the fields of biomedicine and material science; for instance, it is used as a composite coating material to harden polymers as it exhibits surface roughness in nanoscale, and their aggregation will create micrometer-sized roughness. Furthermore, their surface energy is very low and can be controlled by anchoring hydrophobic molecules or grafting with polymers [95]. Although silica nanoparticles are hydrophilic, their surface can be modified with a material having low surface energy; thus, they are popularly used to fabricate materials with a superhydrophobic surface [96].  $\text{SiO}_2$  displays unique

properties including high hardness, low refractive index [97-99], excellent intrinsic characteristics such as nontoxicity, high thermal and mechanical stability, and easy structural regulation [100] making it one of the most researched and applied compounds in protective coatings [101]. The adhesives include epoxy resin [102-104], methyl phenyl silicone resin [105], benzoxazine [106], hydroxyl acrylic resin [107], commercial spray adhesives, double-sided tape [108]. The fabrication process involves forming either a bilayer coating containing top micro/nanoparticles layer and bottom binder layer or a monolayer coating composed of adhesive and other materials such as nanoparticles and solvents.

## Mechanism of the Sol Gel Process

The sol-gel process offers a convenient method to fabricate porous glass films at ambient conditions because it is versatile, cost-effective and easy to combine with liquid deposition techniques that permit an accurate control of the coating thickness [99]. In a typical sol-gel process, the precursor is converted into a glassy material through a series of hydrolysis and polycondensation reaction. The most important factor for the precursor solution that influences the sol-gel process is the catalyst [109]. Referring to the chemistry of alkylsilanes sol-gel, it is well-known that the silica phase separation easily takes place when the pH value of the mixing solution is below and above the isoelectric point (IEP) of the reacting silanol. The formation of linear structures results from the acid catalyst reaction, while coagulation phenomena occur at base catalyst conditions [12]. It has been reported in the literature that incorporation of nanoparticles and microparticles in sol-gel or organic-inorganic hybrid or polymer matrix can lead to superhydrophobicity [110,111]. Silica nanoparticles have also been used for the preparation of superhydrophobic surfaces after hydrophobization with low surface energy materials like octadecyltrichlorosilane, polydimethylsiloxane or perfluoroalkyl methacrylic copolymer [112,113]. Generally, there are two superhydrophobic states on a rough surface: Wenzel's state [114] and Cassie's State [115]. The former represents a wet contact mode of water and rough surface, where water droplets pin the surface and cannot slide on the surface. The latter represents a non-wet contact mode and water droplets can roll off easily owing to their low adhesive force [3]. Wenzel and Cassie theories [115-118] have been widely adopted in correlating the water contact angle (WCA) and surface textures and thus interpreting most of the experimental results. A combination of dual

scale structure, i.e., surface roughness and low surface free energy results in artificial superhydrophobic surfaces. Attempts have been made in fabricating and mimicking such surfaces applying coatings with high WCA [119,120].

## Literature Review

Some literature discusses the development of fabricating transparent superhydrophobic surfaces [121-123]. Some authors [109,124,125] reported about the synthesis of artificial superhydrophobic surfaces [52,126-131] by using a dispersion of silica (or titanium dioxide) nanoparticles to create an appropriate surface roughness and a fluorinated cover layer to modify the surface chemical nature [12]. Carla, et al. [132] demonstrated that chemical structure played a significant role on the topography and average roughness, so the roughness value of 125~150nm was necessary for the formation of superhydrophobic surfaces. Different hydrophobic organosilane compound has been adopted for preparing superhydrophobic silica coating in combination with tetraethylorthosilicate (TEOS) [133-136]. Cai, et al. [137] and Wang and Xiong [138] used methyltriethoxysilane (MTES) and trimethylethoxysilane (TMES) as precursors to obtain hydrophobic antireflective coatings, respectively. Some other researchers used a cationic surfactant to regulate surface morphologies, Meng, et al. [139] used trimethylchlorosilane (TMCS) as surface modification agent, Gurav, et al. [140] used methyltrichlorosilane (MTS) and Purcar, et al. [141,142] used diethoxydimethylsilane(DEDMS) and hexadecyltrimethoxysilane and tetramethoxysilane (C16TMS/TMOS). In this section we give a brief survey about most of the published trials to fabricate superhydrophobic surfaces;

Alamri et al [52] develop an alternative, approach for the synthesis of a superhydrophobic magnetic-core/silica-shell-grafted nanoparticles with superior and durable superhydrophobic performance under a variety of corrosive conditions. An, et al. [143] revealed that mountain-like protrusions appeared on the coated superhydrophobic cotton fabric surface after adding SiO<sub>2</sub> particles into the fluorinate-containing polyacrylate, could capture more air for the WCA increase. The polyacrylate/SiO<sub>2</sub> hybrid coatings not only show good water repellency but also exhibit excellent mechanical properties. Aytug, et al. [144] fabricated a unique three-dimensionality interconnected nanoporous coatings, which significantly suppresses Fresnel light reflections and provide enhanced transmission. Barkhudarov, et al.

[145] prepared superhydrophobic films using fluoroalkylsilanes and studied their effectiveness as corrosion inhibitor using neutron reflectivity technique. Basu et al [41] fabricate superhydrophobic surface by applying precursor mixtures containing hydrophobically modified silica (HMS) nanoparticles dispersed in sol-gel matrices on different substrates by spray and spin coating. Bravo, et al. [146] created transparent superhydrophobic films based on silica nanoparticles through a layer-by-layer method, calcinated at 550 °C, otherwise, films would be removed from the substrate by rubbing with an alcohol-soaked wipe. Budunoglu, et al. [147] reported a thermally stable flexible and highly porous aerogel thin films having superhydrophobic properties using a colloidal dispersion of an intrinsically hydrophobic organosilane monomer at ambient conditions. Caldona, et al. [148] constructed superhydrophobic and superoleophilic nanocomposite film by incorporating SiO<sub>2</sub> nanoparticles into the rubber modified polybenzoxazine by dipping and spraying technique [33]. Chen, et al. [36] fabricate a superhydrophobic and durable coating, through the synthesis of non-fluorine siloxane solution including tetraethoxysilane (TEOS), 3-(methacryloyloxy)propyltrimethoxysilane, and hexadecyltrimethoxysilane (HTMS) via a facile one-pot method.

Cholewinski, et al. [149] reported a robust superhydrophobic bilayer coating containing polydimethylsiloxane (PDMS)-functionalized silica particles on top and an epoxy bonding layer at the base of the substrate. Ding et al [150] fabricated a superhydrophobic wood surface by casting a superhydrophobic coating, prepared by blending TiO<sub>2</sub> nanoparticles with, w-bis (hydroxypropyl)-terminated fluorinated polysiloxane oligomer (PMSF) and, ω-bis (hydrogen)-terminated poly(dimethylsiloxane) solutions. Ebert and Bhushan [151] prepared transparent superhydrophobic surfaces with a dip coating solution composed of surface-functionalized nanoparticles, tetrahydrofuran/isopropyl alcohol solvent and methylphenylsilicone resin, and the obtained surfaces showed wear resistance for potential commercial use in sliding and water jet experiments. Gao and McCarthy [152] prepared a perfectly hydrophobic surface with a water contact angle of more than 170° on a silicon wafer by submerging it in a toluene solution of methyltrichlorosilane (MTCS) at room temperature. Goswami, et al. [11] present a simple, inexpensive sol-gel dip coating procedure which is based on the preparation of a macroscopic network with silica gel nanoparticles (NPs) through the hydrolysis of silicon alkoxide and

condensation of silanols. Guo and Wang [18] develop an efficient one-step method to fabricate superhydrophobic silica-based polymer coatings by adding modified SiO<sub>2</sub> nanoparticles through polymer matrix, and reported that the superhydrophobicity is well maintained in acidic and basic solutions, and this method possesses the advantages of being both simple and inexpensive as well as utilizing non-fluorine-containing compounds, which may bring great advantages in industrial applications, moreover, water repellency of the coating surface can be dramatically enhanced. Guo, et al. [3] fabricated a superhydrophobic surface-modified SiO<sub>2</sub>/nylon 6,6 nanocomposite coating based on hydrophilic nylon 6,6. The adding of surface-modified SiO<sub>2</sub> nanoparticles affected the coating surface energy and surface roughness, in turn influencing the wettability of the coating. Gurav, et al. [101] offer an effective and simple method for preparing self-cleaning superhydrophobic coatings on glass substrates, where the silica particles were easily modified by methyl groups using MTCS as a modifying agent; then applied to a glass substrate using a simple dip-coating technique.

Hsieh, et al. [153] produced superhydrophobic fluoro-containing silica coatings with good superhydrophobicity and superoleophobicity for sunflower oil on a wood substrate by spraying a mixture of perfluoroalkyl methacrylic polymer solution with SiO<sub>2</sub> nanoparticles. Hu, et al. [33] fabricate superhydrophobic and mechanically robust nanostructured films by modification of SiO<sub>2</sub> nanoparticles with hexamethyldisilazane (HMDS) and the subsequent deposition of films by dip-coating a mixture of the as-synthesized superhydrophobic SiO<sub>2</sub> nanoparticles, the hydroxyl acrylic resin (HAR) and the commercial SiO<sub>2</sub> nanoparticles. Huang and Lin [154] fabricated superhydrophobic transparent surfaces by a sol-gel method with a silica-based coating in ethanol and subsequent coating of a low surface energy material like 1H,1H,2H,2H-perfluorooctyltrichlorosilane. Huang, et al. [155] developed a superhydrophobic paper via mixing MPS-modified TiO<sub>2</sub> nanoparticles modified by silane coupling agent, 3-(trimethoxysilyl) propylmethacrylate (MPS), into cellulose pulp, after which the pulp mixture was diluted with distilled water to prepare hand sheets through a series of basic operation including vacuum filtration, dehydration, and curing. Huang, et al. [156] synthesized the fluorinated acrylate copolymers by solvent polymerization using ethanol and methyl ethyl ketone as solvents, and then the silica/fluorinated acrylate copolymers hybrid films were prepared by mixing silica sol and fluorinated acrylate copolymers. The hybrid films had good hydrophobicity, high thermal



stability and excellent mechanical properties of adhesion strength and pencil hardness. Hwang and Ahn [96] reported about the fabrication of a superhydrophobic surface using silica nanoparticles through the chemical reaction of the hydroxyl groups on the silica surface with aminopropyltriethoxysilane (APTES), followed by the condensation of palmitoyl chloride with the modified nanoparticles. Jiang and Guo [157] prepared robust superhydrophobic coating by spreading tungsten oxide microparticles onto epoxy resin treated glass substrates. Kamitani and Teranishi [158] have compared surfaces coated with fluoroalkylsilanes and polydimethylsiloxanes and reported that water contact angle of the surface coated with fluoroalkylsilanes was higher than that with polydimethylsiloxanes, but the sliding angle (SA) of the surface coated with fluoroalkylsilanes was higher than that with polydimethylsiloxanes [41]. Ke, et al. [10] develop polydimethylsiloxane (PDMS) based superhydrophobic surface by drop-coating a mixture of silica and PDMS on the glass substrate. Khalilabad and Yazdanshenas [159] demonstrated a dip-pad-dry coating method for depositing graphene oxide on cotton fibers. When combined with a subsequent reaction with MTCS, the method yielded polymethylsiloxane (PMS) nanofilaments on the fiber surface, which converted highly water-absorbing insulator cotton fabrics into textile-based substrate conductors with superhydrophobic properties. Khoo and Tseng [160] described in detail the hydrophobic engineering of three-dimensional nano-architectures with various shapes, morphologies, and sizes using the phase separation of MTCS on glass and silica substrates. Kumar, et al. [161] reported a self-cleaning glycidoxypropyltriethoxysilane coatings with 15 wt.% loading of 10-20 nm silica by using organic-inorganic hybrid sol-gel method. Lakshmi, et al. [162] fabricated superhydrophobic sol-gel nanocomposite coatings by incorporating silica nanoparticles in an acid-catalyzed ethanol-water mixture sol of methyltriethoxysilane (MTEOS). The water repellency was further improved by the addition of fluoroalkylsilanes.

Latthe, et al. [133] develop semi-transparent superhydrophobic silica-polymethylmethacrylate (PMMA) coatings on the glass by using a hybrid sol-gel method. Li and Shen [163] designed a two-step dipping process with a silica sol and silica microsphere suspension for the preparation of superhydrophobic surfaces with a water contact angle of 170°. Li, et al. [164] fabricated a superhydrophobic polymer coating by a facile phase separation method through spraying the fluorinated acrylic random copolymer micelle solution. Liang, et al.

[165] developed a facile sol-gel method to create a superhydrophobic surface with tetraethylorthosilicate (TEOS) and vinyltriethoxysilane (VTES) as co-precursor following the typical Stöber method. Liu et al [7] investigated a fabrication of polyvinyl alcohol (PVA)/silica (SiO<sub>2</sub>) composite polymer coating on wooden substrates with super repellency toward the water, low sliding angles, low contact angle hysteresis, and relatively better mechanical robustness. They reported that the composite polymer slurry, consisting of well-mixing SiO<sub>2</sub> particles and PVA, is prepared simply and subsequently coated over wooden substrates with good adhesion. The SiO<sub>2</sub> particles act as a solid plasticizer capable of enhancing the chemical and mechanical properties and improve the dimensional stability of the PVA/SiO<sub>2</sub> composite polymer [166]. Liu, et al. [167] fabricated a superhydrophobic vinyltriethoxysilane (VTES)- modified silica coating on a wood surface using a one-step hydrothermal method. Liu, et al. [168] took candle soot as an intermediate to fabricate superhydrophobic and transparent surfaces on glass. Luo, et al. [169] fabricated an almost transparent polyurethane/silica composite superhydrophobic coating by a double-step sol-gel route, which improved the dispersion of SiO<sub>2</sub> particles obviously. Mahadik, et al. [24] presented the combined approach to fabricate recoverable superhydrophobic surface, more in line hierarchical texture with low energy surface used to fabricate superhydrophobic surface. This approach involves a direct growth of dual scale silica particles using dip coating deposition technique and surface energy reduction by surface modification with trimethylchlorosilane (TMCS). After necessary surface modification, a highly thermally stable, mechanically robust and recoverable superhydrophobic surfaces have been fabricated. Manca, et al. [12] generate robust superhydrophobic surfaces exhibiting a self-cleaning behavior by simply depositing a sol-gel-based coating containing trimethyl siloxane (TMS) surface-functionalized silica nanoparticles onto a pre-coated silica gel layer. Also, they [12] reported on the synthesis of superhydrophobic antireflective coatings using a bilayer approach through the sol-gel process. They used trimethylsilanized silica nanoparticles (Aerosil) partially embedded into an organosilica binder matrix and claimed that the cured coatings at 350°C have retained the superhydrophobicity even after 2000 h of outdoor exposure [11].

Martin and Bhushan [105] also prepared the transparent, wear-resistant, superhydrophobic and superoleophobic PDMS/binder surfaces by spraying a mixture of hydrophobic silica nanoparticles,

tetrahydrofuran, isopropyl alcohol and methyl phenyl silicone resin with subsequent deposition of fluorosilane, where the PDMS substrate was required to be chemically activated by ultraviolet-ozone treatment. Ogihara, et al. [170] fabricated superhydrophobic and transparent silica nanoparticle films on glass plates by spray coating suspensions containing 1-propanol and hydrophobic nanoparticles that were modified with dodecyl groups. Pilotek and Schmidt [110] have obtained sol-gel hydrophobic coatings by dispersing silica nanoparticles in a matrix containing tetraethylorthosilicate (TEOS) and fluoroalkyl silane, then investigate the wettability of these microstructured coatings [41]. Prakash, et al. [171] reported the preparation of silica aerogel thin films at ambient pressure without the need of supercritical extraction. Prasad, et al. [172] prepare transparent hydrophobic and superhydrophobic polyamide12-SiO<sub>2</sub> nanocomposite transparent coatings on the glass surface. The coatings have been prepared through the successive spin coating of hydrophobically modified silica (HMS) dispersed onto polymer films. Psarski et al [104] developed superhydrophobic coating via incorporating glass microbeads and Al<sub>2</sub>O<sub>3</sub> nanoparticles in epoxy resin with subsequent surface sandblasting and wet chemical hydrophobization. Ramezani, et al. [173] studied the two-step dip coating via a sol-gel process to prepare superhydrophobic silica films by a coating of silica alcohol and subsequent modification with isoctyltrimethoxysilane as a hydrophobic agent. Rao and his coworkers [134,174] have prepared superhydrophobic silica film by introducing methyl groups in the silica film by post-synthesis grafting from solutions using trimethylchlorosilane (TMCS) and HMDS silylating agents [41]. Shang et al [16] reported about fabrication of superhydrophobic surface with core/shell SiO<sub>2</sub>/ fluoro-containing polymer (SiO<sub>2</sub>/PFS) composite particles via one-step emulsion polymerization and the drop-coating process has been presented. The as-prepared superhydrophobic SiO<sub>2</sub>/PFS coating has a water contact angle up to 171° mainly due to the combination of the hierarchical micro/nano-scale roughness and the low surface free energy. Shang, et al. [175] prepared superhydrophobic silica coatings by spraying an ethanol suspension consisting of silica sol and silica microspheres followed by hydrophobic treatment with 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDTs) solution. Shang, et al. [176] have developed optically transparent superhydrophobic silica-based films by means of sol-gel processing and self-assembly. The resultant silica-based coatings showed advancing and receding water contact angles of approximately 165/115 respectively, and hence the water drops do not roll on such coatings due to the

high contact angle hysteresis. Shang, et al. [175] reported a simple, versatile strategy for creating a superhydrophobic surface via spraying the as-prepared silica suspension containing silica sol and silica microspheres on the substrate, using 1H,1H,2H,2H-perfluorodecyltriethoxysilane to form hydrophobic groups.

Shi and Xiao [177] fabricate superhydrophobic nanocomposite coating by spraying nanocomposite dispersion containing hydrophobic silica nanoparticle and polyvinylidene fluoride (PVDF) on the paper surface. Shirgholami, et al. [178] recently reported the deposition of polymethylsilsesquioxane (PMSQ) nanostructures onto the surface of the cotton fabric and adopted the subsequent modification in a toluene solution of MTCS at room temperature with different duration times to achieve superhydrophobic cotton fabrics. Song, et al. [179] proposed the fabrication of hydrogel and polymeric spheres over a superhydrophobic substrate for potential application in tissue engineering or as support for cell expansion. Su, et al. [19] fabricated organic-inorganic superhydrophobic polyester textiles with strong durability and robustness for practical applications in oil spill accidents and industrial sewage emission via a very simple approach with cheap tetraethylorthosilicate (TEOS) and polydimethylsiloxane (PDMS (OH) as reactants using HCl as catalysts. The textile possessed excellent water repellency with a contact angle of 160°. The formation process of the superhydrophobic surface included the first hydrolysis-condensation of TEOS to generate silica, the subsequent gradual formation of micro-aggregated silica to construct roughness under the driving force of the extreme polarity difference between silica and PDMS(OH), and the further crosslinking reaction of Si-OH groups between micro-aggregated silica and PDMS(OH) at the later reaction stage. Su, et al. [180] have embedded nano silica particles on epoxy coated glass surface and subsequently coated with fluoroalkylsilane to achieve superhydrophobicity. Fluoroalkylsilanes are either incorporated in the hybrid mixture or applied as a topcoat to decrease the surface energy [110,124,180,181]. Sun, et al. [182] constructed titanium dioxide nanoparticles (NPs) on a wood surface through a hydrothermal method and then modified the rough structure with sodiumdodecylsulfate (SDS), to yield an apparently superhydrophobic wood surface with a water contact angle of about 154°. Tang, et al. [183] fabricated a transparent superhydrophobic coating on paper by dip coating paper samples in silica ethanol sol through co-hydrolysis of TEOS and hexadecyltrimethoxysilane (HDTMS), followed by curing coating overnight at indoor

temperature. Tu, et al. [184] prepared superhydrophobic coating by spraying a waterborne perfluoroalkyl methacrylic copolymer emulsion mixed with 50 wt.% TiO<sub>2</sub> nanoparticles on polydimethylsiloxane pre-coated wood. Wang and Xiong [138,185] prepared two different rough coatings and compared the influence on the transmittance and wettability. The wettability on the surface with micro-/nano-hierarchical structure was relatively stable with respect to the surface with low roughness. And he indicated that the air bubbles trapped in the surface were also an important factor to influence the hydrophobicity. Wang, et al. [186] synthesized silica particles on wood surfaces via a sol-gel process and subsequently modified hydrophilic particles with octadecyltrichlorosilane via a chemical vapor deposition (CVD) method to finally obtain a superhydrophobic wood surface with a water contact angle of 164°. Wang, et al. [187] fabricated zinc oxide nanorods on a wood surface using a wet chemical approach followed by modification with stearic acid, thereby endowing the wood surface with superhydrophobic properties and yielding a water contact angle value of about 153.5°.

Wang, et al. [188] developed a novel transplantable superhydrophobic film on a pretreatment glazed tile by spraying 1H,1H,2H,2H perfluorodecyltriethoxysilane solution with 32.5 wt.% SiO<sub>2</sub>. Wei, et al. [189] presented a simple way to prepare a superhydrophobic thin film which was composed by one-step coating with modified silica nanoparticles. Wu, et al. [190] have developed transparent superhydrophobic hard coatings by sol-gel method with silica filler resulting in rough surfaces with a lotus-like structure but the obtained water contact angle was about 123° [191]. Wu, et al. [192] produced durable superhydrophobic surfaces by spraying, dipping and brushing emulsion composed of nanoparticles and epoxy resin on different substrates. Xing et al [5] synthesize various nano-coatings with different particle stacking manner (i.e., micro- and/or nano-roughness) by using a series of dendrimer-like porous silica nanospheres (DPSNs) with tunable particle sizes and surface pore sizes as building blocks, based on paint- and dip-coating methods. Xu, et al. [193] fabricated a hierarchically structured lotus-leaf-like superhydrophobic films by spraying the polystyrene/SiO<sub>2</sub> core/shell nanoparticles as a coating skeleton and the polydimethylsiloxane as hydrophobic interconnection and reported average transmittance greater than 95% and a water contact angle of 162°. Xue, et al. [194] fabricated a superhydrophobic textile with durability against abrasion and laundering through three steps including the creation of rough structure by alkali etching, modification with mercapto

siloxane and hydrophobization with fluorine-containing materials. Yang and Deng [195] fabricated superhydrophobic surfaces on paper by multi-layer deposition of polydiallyldimethylammonium chloride (PDADMAC) and silica particles, after which the samples were modified by 1H,1H,2H,2H-perfluorooctyltriethoxysilane (PFOTS). Zhang and his coworkers [196] fabricated micro-nano hierarchically structured nylon 6,6 surfaces by a phase separation method and the wettability of the surfaces changed from super hydrophilicity to superhydrophobicity after further modification by low surface energy material. Zhang and Seeger [197] successfully prepared a superhydrophobic and superoleophilic textile as a membrane for oil-water separation and selective oil absorption. Zhang, et al. [40] proposed a feasible approach to fabricate a durable superhydrophobic coating with litchi-like structure by evenly growing nano-SiO<sub>2</sub> particles on polyethersulfone (PES) submicron particles. Zhang, et al. [198] fabricated novel nanocomposite coatings with the transmittance values of 96.86%~97.34%. They pointed out that the high porosity is not good for the transmittance and durability, and furthermore he got a suitable refractive index of 1.21 by controlling the porosity size. Zhao, et al. [199] synthesize silica coated multiwalled carbon nanotube (MWCNTs) composites (SiO<sub>2</sub>/MWCNTs) by means of the sol-gel method. They reported that the surface energy of materials was greatly reduced due to modifying SiO<sub>2</sub>/MWCNTs with vinyltriethoxysilane, in addition to the rough structure of carbon nanotubes. Consequently, superhydrophobic surface with maximum static water contact angle of 156° and sliding angle of 3.5° was fabricated.

Zhao, et al. [200] prepared a superhydrophobic coating solution via co-hydrolysis and condensation of TEOS and a fluorinated alkylsilane under the alkaline condition on various substrates such as cotton and filter paper. The superhydrophobic coatings exhibited good transparency, which the water contact angle of 170° and a sliding angle of no more than 10°. Zhong, et al. [201] developed transparent and durable superhydrophobic bilayer coatings containing hydrophobic silica particles on top and an epoxy resin bonding layer at the base. Hydrophobic silica nanoparticles prepared by using the co-precursors of tetraethylorthosilicate (TEOS) and methyltriethoxysilane (MTES), and then highly dispersed in ethanol to form a brush-coating suspension. They concluded that the stability of the coating decreases with decreasing of silica concentration. Zhou, et al. [202] reported a durable and robust superhydrophobic textile coating by mixing polydimethylsiloxane (PDMS) with

fluorinated alkylsilane (FAS) functionalized silica nanoparticles and FAS. Zhou, et al. [203] prepared a superhydrophobic polydopamine-coated copper foam for oil/water separation by utilizing the self-polymerization of dopamine to enhance the interface bonding strength between the copper foam and Ag nanoparticles. Moreover, stable Fe<sub>3</sub>O<sub>4</sub>-PDA-Ag nanoparticles with core-shell hierarchical structures were prepared via step wisely cladding dopamine and silver particles on the surfaces of Fe<sub>3</sub>O<sub>4</sub> [204].

## Conclusion

Fabrication of superhydrophobic surfaces is crucial for commercial applications of these surfaces in the fields of self-cleaning, water repellency and corrosion resistance. A sol-gel method is a simple approach for the fabrication of the superhydrophobic silica coating, where surface modification of the SiO<sub>2</sub> and the curing temperature are very important for obtaining stable superhydrophobic surfaces. The hydrophobicity of the surface depends on the thickness of the deposited films. These hydrophobic coating materials alter surface wettability from hydrophilic to hydrophobic one. The enhancement of the mechanical properties can be attributed to the fact that the SiO<sub>2</sub> nanoparticles act as a solid plasticizer capable of enhancing the chemical and mechanical properties, and enhance the stability of the water contact angle of the composite coating.

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