CHAPTER 4

Nanomaterials for design and fabrication of superhydrophobic polymer coating

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1. Introduction

Superhydrophobic materials are highly hydrophobic and it is extremely difficult to wet such surfaces [1]. The water contact angle of superhydrophobic materials may exceed 150 degrees. Superhydrophobic surfaces have been used for numerous engineering applications such as self-cleaning, antibiofouling, anticorrosion, biomedical, and textiles [2]. Significantly protecting properties of the coatings have been achieved using superhydrophobic materials. Successful fabrication of superhydrophobic surfaces demands controlled generation of hierarchical rough morphology [3]. The choice of hydrophobic polymers such as fluoropolymers, perfluorooctanoic acids, etc. is also important to form superhydrophobic surfaces [4, 5]. Superhydrophobic polymer morphology and roughness can also be altered using nanoparticles, lithography, etching, biomimetic, and stamping processes [6]. The superhydrophobic surfaces may have additional features such as magnetic, mechanical, wear, thermal, conducting, etc. [7–10]. The hydrophobic polymers have been reinforced with various nanoparticles (carbon nanotube (CNT), graphene, carbon nanofiber, carbon black (CB), zinc oxide, silica, etc.) to form superhydrophobic materials [11–13]. Nanocarbon-based superhydrophobic surfaces are low cost having high electrical and thermal conductivity and strength. The multifunctional polymer/nanocarbon superhydrophobic surfaces have been employed in electronics, electromagnetic interference (EMI) shielding devices, strength and wear demanding materials, and biomedical. In this chapter, progress in the field of polymer/nanocarbon, polymer/inorganic nanoparticle, and polymer/hybrid organic-inorganic nanoparticle for superhydrophobic coatings is comprehended. Initially, basics of superhydrophobic coatings and nanocomposite coatings have been outlined. Finally, future opportunities and challenges in the emerging technological field of superhydrophobic coatings have been discussed.

Superhydrophobic Polymer Coatings
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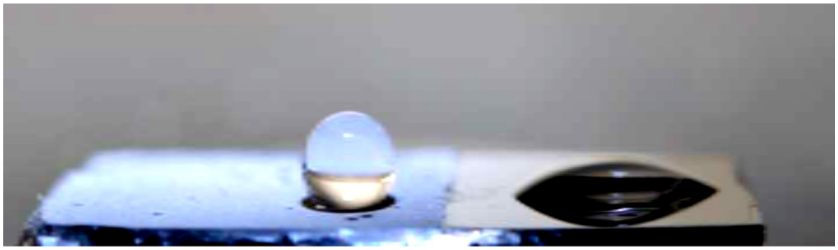

Fig. 1 Superhydrophobic coating. *(Reproduced with permission from Ovaskainen, L., et al., Superhydrophobic polymeric coatings produced by rapid expansion of supercritical solutions combined with electrostatic deposition (RESS-ED), J. Supercrit. Fluids 95 (2014) 610–617, Elsevier.)*

2. Superhydrophobic polymer coating

Superhydrophobic surfaces have gained interest owing to excellent water repulsion and self-cleaning properties (Fig. 1) [14]. This effect is commonly known as "lotus effect." The properties of lotus leaf have been mimicked using micro- and nanoscale materials [15, 16]. Good superhydrophobic surfaces must have water contact angle >150 degrees. On the other hand, contact angle hysteresis and roll-off angle must be low <10 degrees. Several polymers have been used to form superhydrophobic surfaces such as polystyrene (PS), poly(vinyl fluoride), poly(methyl methacrylate), epoxy, and several other vinyl and acrylic polymers. Spin coating, electrospraying, electrodeposition, electrospinning, etc. have been used to form superhydrophobic surfaces [17]. The superhydrophobic coating of PS was prepared using electrospinning process and dimethylformamide (DMF) solvent. The water contact angle of >150 degrees was obtained [18]. Superhydrophobic surface of poly(hydroxybutyratecohydroxyvalerate) (PHBV) and chloroform has been prepared using electrospinning [19]. The PHBV surface had water contact angle of 76 degrees. The surface roughness and water contact angle were increased up to 158 degrees. The dispersion of polytetrafluoroethylene nanoparticles in poly[tetrafluoroethylene-*co*(vinylidenefluoride)-*co*-propylene] was prepared using electrospinning and water contact angle of >150 degrees was attained [20]. Ovaskainen et al. [21] produced superhydrophobic polymeric coatings using rapid expansion of supercritical solution (RESS) and electrostatic deposition (ED). Poly(vinyl acetate)poly(vinyl pivalate) was dissolved in supercritical carbon dioxide and acetone. The mixture was sprayed through a nozzle with an applied voltage of 8 kV. Fig. 2 shows setup for spraying technique used for ED, that is, RESS-ED. Processing parameters were well controlled using RESS-ED technique and the superhydrophobic surface produced with spraying was excellent water repellent. Moreover, RESS-ED process may yield large, fine, and thin coatings.

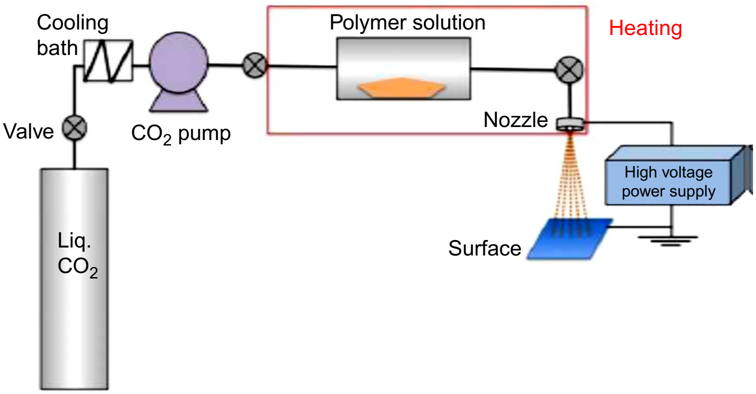

Fig. 2 Schematic diagram of RESS-ED setup used in spraying experiments. *(Reproduced with permission from Ovaskainen, L., et al., Superhydrophobic polymeric coatings produced by rapid expansion of supercritical solutions combined with electrostatic deposition (RESS-ED), J. Supercrit. Fluids 95 (2014) 610–617, Elsevier.)*

3. Superhydrophobic polymer nanocomposite coating

Carbon nanomaterials have been focused for superhydrophobic coatings on metals, polymers, wood, or textile [22, 23]. Nanocarbon such as CB, graphene, and CNT is water repellent agents [24]. These nanocarbon allotropes can be structured at micro/nanoscale to gain superhydrophobic properties. Vertically aligned CNT arrays or CNT forests have been used to imitate water repellent properties of lotus leaves [25]. Carbon nanomaterials (graphene (Gr) and CNT) have been deposited on rough wood surface to create superhydrophobicity. Simple drop casting and dip coating methods have been used to form carbon nanomaterials [24]. The contact angle measurements of wood coated material were >130 degrees. The Gr and CNT were physically bonded to the wood surface. Fig. 3 shows a proposed model for carbon nanomaterial deposition on wood. The surface is partly wetted by water. The superhydrophobic effect was observed due to micro-

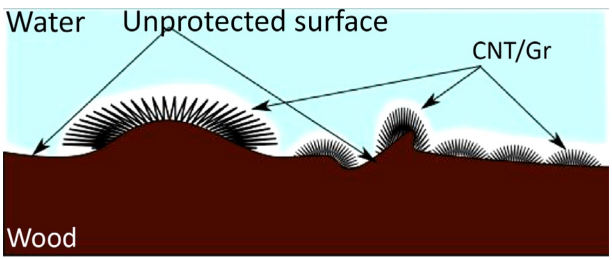

Fig. 3 Cassie's impregnating state model for wood covered with carbon nanomaterial. *(Reproduced with permission from Łukawski, D., et al., Towards the development of superhydrophobic carbon nanomaterial coatings on wood, Prog. Org. Coat. 125 (2018) 23–31, Elsevier.)*

roughness of the wood surface. The CNT/H_2O/BW and Gr/H_2O/BW (CNT = carbon nanotube; Gr = graphene; and BW = balsa wood) became superhydrophobic (contact angle > 130 degrees) after surfactant removal (Fig. 4A). The contact angle was stable after 10 min for CNT/H_2O/BW, that is, 134 degrees (Fig. 4B). Moreover, the deposited droplets were steadfastly attached to the surface (Fig. 4C). On the other hand, the presence of surfactant render the coatings hydrophilic (Fig. 4D). Using polymeric nanocomposite hydrophilic wood surface has been successfully transformed into the superhydrophobic materials. The superhydrophobic nanocomposite coatings of polyethylene terephthalate/multiwalled carbon nanotube (MWCNT)/silica nanoparticles were fabricated using spray method [26]. The nanocomposite coating with 0.2 wt% MWCNT had excellent hydrophobicity with contact angle of 156.7 degrees. There was 95.7% transparency and sheet resistance of $3.2 \times 10^4\,\Omega\,\text{sq.}^{-1}$. Superhydrophobic self-cleaning nanocomposite coating of polytetrafluoroethylene/TiO_2 has been reported [27]. The superhydrophobic polymer/tungsten oxide (WO_3) nanocomposite coatings were developed on glass substrates [28, 29]. Thus, various designs and compositions of polymer nanocomposite coatings have shown unique chemical structure, enhanced superhydrophobicity, and highly rough surface to be employed in relevant applications.

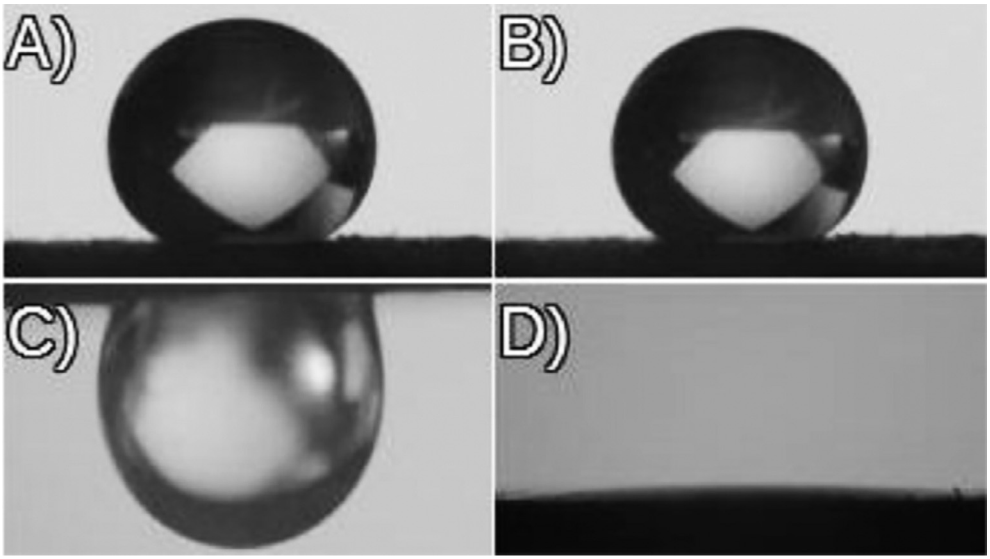

Fig. 4 A droplet of water at CNT/H_2O/BW (A) immediately after dropping; (B) 10 min after dropping; (C) with surface turned upside-down; and (D) without surfactant rinsing. *(Reproduced with permission from Łukawski, D., et al., Towards the development of superhydrophobic carbon nanomaterial coatings on wood, Prog. Org. Coat. 125 (2018) 23–31, Elsevier.)*

4. Polymer/nanocarbon nanocomposite coating

4.1 Polymer/nanodiamond-based superhydrophobic coating

Nanodiamond (ND) is a unique nanocarbon having exclusive surface properties, electronic and optical features, hardness, and biocompatibility useful for polishing technology, coatings, catalysis, drug carriers, cosmetics, etc. [30, 31]. Polymer and ND core-shell particles have been prepared with superior surface roughness, efficient light scattering, thermal conductivity, and superhydrophobicity [32, 33]. Physical or chemical adsorption of polymers on ND using intermolecular interaction such as ionic interaction/hydrogen bonding may form stable superhydrophobic structures. ND is a promising nanocarbon material used in various applications demanding superhydrophobicity. Takafuji et al. [34] demonstrated superhydrophobic thin ND layer by mixing polymer particles. Cao et al. [35] prepared superhydrophobic coating of hydroxylated ND, polydopamine (PDA), and 1*H*,1*H*,2*H*,2*H*-perfluorodecanethiol (PFDT). The PDA modified ND nanoparticles were anchored on commercial polyurethane (PU) sponge. The sponge was used for the selective removal of oil from water. Fig. 5 shows the absorption capacity of superhydrophobic ND coated PU sponge. The foam was tested for diesel, pump oil, and gasoline over 10 consecutive cycles. The sponge was found highly stable with a variation of 10 cycles. Future research on polymer/ND films or sponges may lead to very

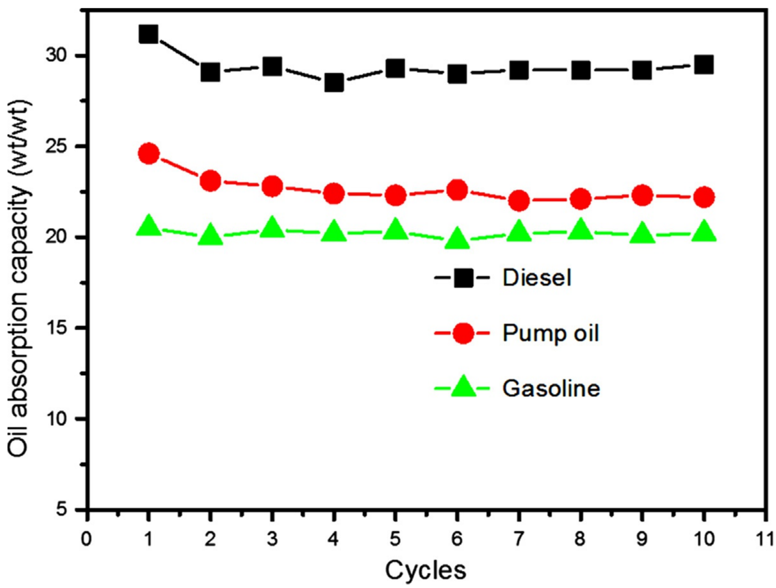

Fig. 5 The oil absorption capacity of superhydrophobic nanodiamond coated polyurethane sponge after 10 cycles of oil removal process. *(Reproduced with permission from Cao, N., et al., Polyurethane sponge functionalized with superhydrophobic nanodiamond particles for efficient oil/water separation, Chem. Eng. J. 307 (2017) 319–325., Elsevier.)*

high organic adsorption capacity and superhydrophobicity. The materials possess low cost, feasible fabrication, and excellent performance toward superhydrophobic properties, oil/water separation, and other desirable practical applications.

4.2 Polymer/fullerene-based superhydrophobic coating

Fullerenes are nanocarbon spheroidal molecules having hollow cage structures of 60 or more atoms, which are often known as buckminsterfullerene [36]. Fullerenes are useful materials in optoelectronics, semiconductors, and energy storage devices [37]. Polymer/fullerene nanomaterials had characteristic properties in electrochemistry, photoelectrochemistry, and hydrophobicity. The C_{60}-porphyrin cocrystals have been self-assembled to form hierarchical architectures [38]. Though, controllable fabrication of fullerene hierarchical structures is challenging [39]. Nakanishi et al. [40] developed superhydrophobic surfaces of polymer and fullerene. Due to π–π and vander Waals interactions between polymer aliphatic chains and fullerene moieties, hierarchical, supramolecular layers were prepared. The superhydrophobic surfaces have high durability. Zheng et al. [41] used drop drying process to form self-assembled superhydrophobic hierarchical fullerene microstructures. The polymer/C_{60} or polymer/C_{70}-based hierarchical structures with superhydrophobicity can be used in high photoluminescence and water-proof optoelectronics.

4.3 Polymer/graphene-based superhydrophobic coating

Research on novel superhydrophobic films having superior self-cleaning, antisticking coating, and oil-water separation has gained attention. Graphene is a single atom-thick nanosheet composed of sp^2-hybridized carbon atom having high hydrophobicity [42]. Fluorinated polymers have high superhydrophobicity and chemical and environmental stability [43]. Poly(vinylidene fluoride) (PVDF) form superhydrophobic surface with low surface roughness and decreased water contact angle. Polymer/graphene nanocomposites have gained increasing consideration owing to improved mechanical properties, electrical conductivity, thermal conductivity, and stimulation-responsive properties [44]. Surface wettability of these materials has been controlled using various graphene nanosheet content [12]. Graphene has been used to enhance the hydrophobicity and roughness to form high-performance superhydrophobic materials [45]. In this regard, PVDF/graphene composites possess superhydrophobicity [46]. Inclusion of small amount of graphene in PVDF led to morphology change as well as significantly enhanced surface roughness. Li et al. [38] developed superhydrophobic hierarchically wrinkled surfaces having high water contact angle of >160 degrees and water sliding <5 degrees. A 500 nm nanostructure was fabricated on PVDF through nanoimprint lithography. Zha et al. [47] formed superhydrophobic PVDF/graphene nanoporous materials. Graphene addition varied the surface properties of superhydrophobic PVDF coatings. Construction of multileveled polymer/graphene structure may reveal critical features of superhydrophobic surface for micro- and nanoscaled devices.

4.4 Polymer/CNT-based superhydrophobic coating

CNT is a nanoallotrope of carbon having cylindrical structure. Caron nanotubes are nanofibrils which may be assembled to form useful structures for technical applications such as electronics, optics, and other materials science relevance [48]. Superhydrophobic CNT materials have been prepared through noncovalent/covalent attachment of nanofiller to hydrophobic polymer molecules [49]. Superhydrophobic CNT layers have been assembled using various techniques such as spray coating, chemical vapor deposition, Langmuir-Blodgett deposition, vacuum filtering, drop casting, etc. [50]. Wang et al. [51] prepared stable superhydrophobic polymer/CNT coatings. The superhydrophobic materials had excellent environmental stability after bending/pressing. Li et al. [52] prepared homogeneous dispersion of an azide copolymer poly(4-azidophenylmethacrylate-*co*-methyl acrylate) and MWCNT in various organic solvents. The π-π interactions were developed between the polymer and nanotube. Addition of MWCNT transformed the material to superhydrophobic with water contact angle of 154 degrees. Yang et al. [53] formed stable and transparent superhydrophobic PS/CNT nanocomposite films using spray casting process. The superhydrophobic films possess high water contact angle of 160 degrees and sliding angle of 38 degrees. The film had light transmittance of 78%. The superhydrophobic polymer/MWCNT films coated on glass, metals, or polymers are highly water repellent, self-cleaning, and having high water contact angles. The polymer/MWCNT-based superhydrophobic coatings may have potential applications in nonwetting surfaces, microfluidic devices, bioseparation, etc.

4.5 Polymer/CB-based superhydrophobic coating

Micro- and nanoscale hierarchically structured surfaces are found vital in producing superhydrophobic surface [54]. CB is a paracrystalline carbon with high surface area-to-volume ratio. Numerous methods have been used to form superhydrophobic surface of polymer and CB such as lithographic patterning, electrodeposition, plasma etching, layer-by-layer assembly, chemical vapor deposition, etc. The CB particles were modified by blending with antioxidant stabilizer to form superhydrophobic film [55]. The performance of polymer/CB composite surface has been enhanced using modified carbon black (MCB). The raspberry-like PS/CB particles have been prepared using heterocoagulaion process through π-π interactions between PS and MCB particles. The heterocoagulation process offered colloid stability in various liquid media [56]. Bao et al. [57] developed raspberry-like PS/CB composite microsphere using heterocoagulation method. Scanning electron microscopy (SEM) was used to study the morphology of raspberry-like PS covered CB particles. The superhydrophobic surface was prepared using the colloidal suspension of PS/CB film. Fig. 6 shows the fabrication process for superhydrophobic particulate film. The MCB dispersion was dropped on PS microspheres under 1020 rpm. The dispersion was further centrifuged at 3000 rpm to remove

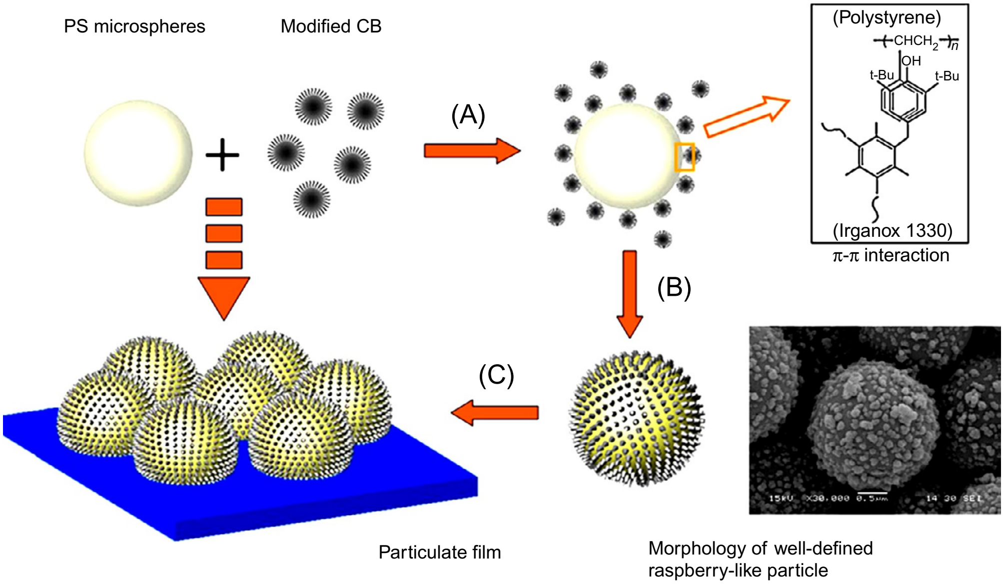

Fig. 6 Schematic illustration for the fabrication of superhydrophobic particulate film: (A) Modified carbon black dispersion on PS microspheres; (B) centrifugation; and (C) superhydrophobic film formation. Inset: SEM photograph of well-defined raspberry-like particles (MCB content 10 wt%). *(Reproduced with permission from Bao, Y., et al., Tailoring the morphology of raspberry-like carbon black/polystyrene composite microspheres for fabricating superhydrophobic surface, Mater. Res. Bull. 46 (5) (2011) 779–785, Elsevier.)*

free CB particles. Finally, superhydrophobic particulate film was deposited. Table 1 reveals the surface properties and root main square (rms) roughness of the particulate film. According to the results, 10 wt% MCB content was found suitable to fabricate the superhydrophobic surface with appropriate surface roughness. The CB-based superhydrophobic surfaces are low cost, scalable, and easy to prepare on commercial scale [58]. The self-cleaning ability of polymer/CB superhydrophobic layers may enable expedient

Table 1 WCA, sliding angle of surface, and rms roughness of relevant raspberry-like particles.

MCB content (wt%)	Water contact angle (WCA) (degrees)	Sliding angle (degrees)	Root main square (rms) roughness (nm)
5	120.3	63.2	16.8 ± 0.7
8	142.5	56.5	19.7 ± 0.6
10	155.2	29.5	28.9 ± 1.0
12	148.0	47.3	23.3 ± 0.8
15	141.4	55.2	17.9 ± 1.4

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recycling process toward various technical applications. The polymer/CB superhydrophobic substrate may have future potential related to solar evaporation.

4. Polymer/inorganic nanoparticle-based superhydrophobic nanocomposite coating

Incorporation of inorganic nanoparticles in polymers enhanced the superhydrophobic nature of coating materials [59, 60]. Superhydrophobic materials based on polymers and inorganic nanoparticles offered large-area, stable, and mechanically robust films on various substrates. The durability of material may be enhanced through cross-linking or covalent bonding between the coating and substrate. Bormashenko et al. [61] developed superhydrophobic surface of disordered triple-scaled arrays of PVDF and metal nanoparticles. The hydrophobicity of inherently wettable surfaces was studied. Liu et al. [62] prepared stable superhydrophobic films of 1*H*,1*H*,2*H*,2*H*-perfluorooctyltrichlorosilane [$CF_3(CF_2)_5(CH_2)_2SiCl_3$] and zinc nanoparticles. The films had effective corrosion resistance when tested in 3%NaCl solution for 29 days. Ebert and Bhushan [63] developed superhydrophobic surfaces using polycarbonate and poly(methyl methacrylate) with surface functionalized SiO_2, ZnO, and indium tin oxide nanoparticles. The surfaces possess high water contact angle, low contact angle hysteresis, high transmission, and good wear resistance. However, fabrication of such surfaces often involves complex or expensive techniques which are not suitable for variety of materials and substrates. Low-abrasion durability is also one of the major issues for polymer/inorganic superhydrophobic coatings.

6. Polymer/hybrid organic-inorganic-based superhydrophobic coating

Organic-inorganic hybrids have been used for superhydrophobic surfaces with fine self-cleaning, antifogging, stability, and biocompatibility properties [64]. For example, incorporation of nanoparticle/microparticle in hybrid polymer matrix may lead to fine superhydrophobicity and other enhanced coating characteristics. Han et al. [65] developed novel organic-inorganic hybrid films of poly(allylamine hydrochloride) and ZrO_2 nanoparticles coated with poly(acrylic acid) using layer-by-layer deposition. Incorporation of hybrid coated ZrO_2 nanoparticles in matrix enhanced the superhydrophobicity and mechanical properties of the organic film. Bayer et al. [66] fabricated nanostructured self-cleaning superhydrophobic polymer/organoclay hybrids. Hybrids of organically modified montmorillonite and acrylic adhesives have been used as superhydrophobic coatings. Organically modified nanoclay in fluoro-methacrylic latex enhanced the abrasion resistant, contact angle, and decreased the hysteresis. Basu and Paranthaman [67] prepared hybrid superhydrophobic surfaces of PVDF/hydrophobically modified fumed silica. With the increase in silica content from 33.3% to 71.4% in PVDF, the water contact angle was enhanced from 117 degrees to 168 degrees. According to SEM, irregular microcavities and nanofilaments on the surface were found responsible for superhydrophobicity and roughness of the hybrid coatings. The method was simple and cost effective for preparing self-cleaning superhydrophobic coatings. Kou and Gao [68] reported silica nanoparticle-coated graphene oxide (GO-SiO_2) nanohybrids. Simple drop-coating method was used. The GO-SiO_2 hybrid had high surface area and semitransparent nature. Adopting new coating methodologies and hybrid materials may pave way for making large surface area hybrid superhydrophobic surfaces. Moreover, the generation of fibrillary hybrid structures and phase separation of polymers may also enhance the surface roughness and superhydrophobicity.

7. Commercial and future prospects and summary

The carbon nanomaterials have natural tendency to transform hydrophilic materials to superhydrophobic. Inclusion of only small wt% of graphene and CNT is enough to acquire waterproofing layer. The coatings can be prepared using drop-casting, dip-coating, spin coating, solution coating, melt coating, blading, chemical vapor deposition, lithography, or other suitable technique to form nanomaterial dispersion. The carbon nanoallotropes, that is, graphene and CNT have enhanced the water contact angle to produce homogenous coatings with long-lasting superhydrophobic effect. According to an estimated value, market price of 1 m^2 coating may be equal 0.02 USD for CNT-based coating and 0.3 USD for CNT-based material. Thus, polymer/CNT nanocomposite may offer high-performance superhydrophobic coatings at very low cost compared with graphene or fullerene-based materials. The CB and CNT-based superhydrophobic surfaces have shown excellent mechanical durability without any additional surface functionalization treatment [69]. Thus, polymer/nanocarbon nanocomposite may offer fine mechanical properties and electrical conductivity in coatings [70, 71]. The carbon nanomaterials act as hydrophobic agents to be employed in polymer sponge, metal mesh, textile, and cellulose materials such as cotton. The microporous structure and low surface energy of carbon nanoallotropes are accountable for superhydrophobicity of coatings. Graphene and nanocarbon materials have been used to enhance the non-wettability of cellulose nanocomposites [72, 73]. Increase in the robustness of superhydrophobic surfaces may bring engineered applications. Nanocarbon-based coatings have high electrical conductivity $\sim$1000 S/m [74]. The synergistic effect of two or more nanocarbon may demonstrate enhanced features for practical applications. The electrical conductivity of superhydrophobic coatings has been used in EMI shielding, enhanced condensation heat transfer, electronics, etc. [75–77]. An important application of superhydrophobic surfaces is oil-water separation [78, 79]. The oleophilic properties of superhydrophobic nanocomposite coatings based on CB, graphene, or CNT have been used to separate out oil from oil/water mixture. The superhydrophobic/oleophilic coatings have been exploited as a filtration membrane for oil/water separator. This chapter presents state-of-the-art discussion regarding superhydrophobic nanocomposite coatings. Organic as well as inorganic nanofillers have been reinforced in polymers to form superhydrophobic surfaces having controlled surface properties, wettability, self-healing, self-cleaning, antifouling, and anticorrosion properties. Moreover, polymeric nanocomposite-based superhydrophobic coating must have improved physical features such as optical, conducting, magnetic, mechanical, and thermal characteristics. High-performance superhydrophobic nanocomposite coatings have been employed in electrical and electronic devices, EMI shielding materials, strengthened materials, and biomedical applications.

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