

## REVIEW ARTICLE

# High-performance functional materials based on polymer nanocomposites—A review

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## ABSTRACT

Oil spill clean-up is a long-standing challenge for researchers to prevent serious environmental pollution. A new kind of oil-absorbent based on silicon-containing polymers (e.g., poly(dimethylsiloxane) (PDMS)) with high absorption capacity and excellent reusability was prepared and used for oil-water separation. The PDMS-based oil absorbents have highly interconnected pores with swellable skeletons, combining the advantages of porous materials and gels. On the other hand, polymer/silica composites have been extensively studied as high-performance functional coatings since, as an organic/inorganic composite material, they are expected to combine polymer flexibility and ease of processing with mechanical properties. Polymer composites with increased impact resistance and tensile strength without decreasing the flexibility of the polymer matrix can be achieved by incorporating silica nanoparticles, nanosand, or sand particles into the polymeric matrices. Therefore, polymer/silica composites have attracted great interest in many industries. Some potential applications, including high-performance coatings, electronics and optical applications, membranes, sensors, materials for metal uptake, etc., were comprehensively reviewed. In the first part of the review, we will cover the recent progress of oil absorbents based on silicon-containing polymers (PDMS). In the later details of the review, we will discuss the recent developments of functional materials based on polymer/silica composites, sand, and nanosand systems.

**Keywords:** polymer; oil-spill; porous materials; composites; silica; nanoparticles

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## ARTICLE INFO

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Received: 5 November 2023

Accepted: 8 December 2023

Available online: 22 December 2023

## COPYRIGHT

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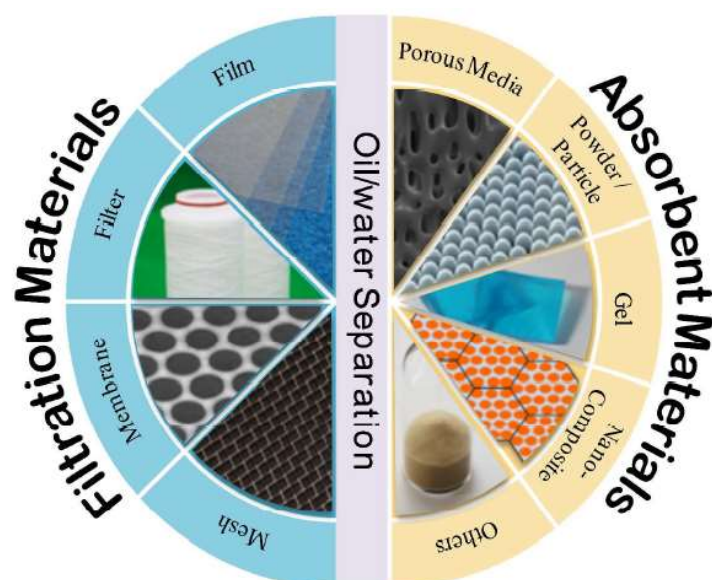
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## 1. Introduction

Various industries, such as mining and petrochemicals, produce enormous volumes of oily water, which has become a ubiquitous pollutant worldwide and is now considered a serious global environmental concern<sup>[1]</sup>. For instance, a typical mining operation produces approximately 140,000 L of oil-contaminated water daily<sup>[2]</sup>. In addition, the frequent oil leakages/spillages during marine transportation or oil production are a potential catastrophe to marine environments and ecology and a tremendous waste of valuable natural resources. Various oil/water separation methods and novel materials have been reported to address these issues<sup>[3]</sup>. The oil-water separation technologies using multiple types of materials are summarized in **Figure 1**.



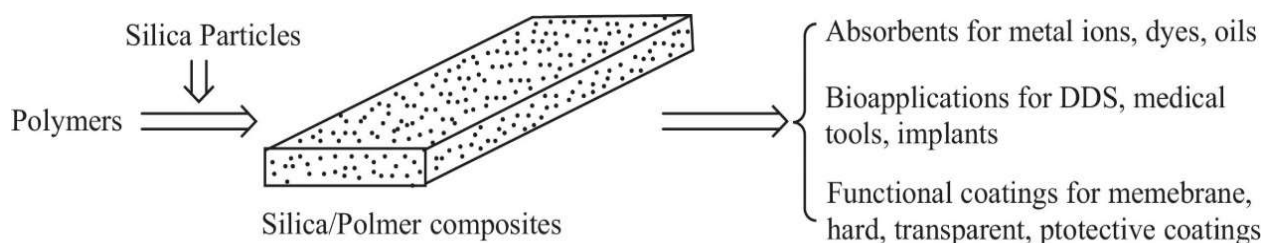
**Figure 1.** The summary of the recent progress of oil/water separation technologies using various materials<sup>[3]</sup>. Reproduced with permission from the Royal Society of Chemistry.

Among the various developed technologies for oil and organic solvent clean-up, the absorption technique has excellent advantages in terms of operational simplicity, cost-effectiveness, and complete clean-up without any secondary pollution<sup>[4,5]</sup>. At present, the oil absorbents are classified into three major categories: (a) porous materials; (b) self-assembled fibers and gels<sup>[6–11]</sup>. Among them, porous materials and polymers with interconnected network structures and swelling properties with considerable high hydrophobicity and oleophilicity are found to be potential candidates for oil absorption due to their outstanding oil selectivity, very high absorption capacities, fast kinetics, excellent material reusability, and enhanced oil recovery<sup>[12–18]</sup>. Recently, PDMS-based absorbents were found to be a potential candidate for oil absorption due to their high hydrophobicity, oleophilicity, and commercial availability<sup>[19]</sup>. In addition, the PDMS has been used to selectively separate oils and/or organic solvents from water selectively<sup>[20]</sup>. The PDMS has been the most widely used silicon-based organic elastomer since Wacker Chemie synthesized the first silicones in the 1950s and its introduction to academic laboratories in the 1990s<sup>[21,22]</sup>. PDMS is generally a viscoelastic, biocompatible, chemically and mechanically robust material with a low glass transition temperature, cost-effectiveness, and good moldability, ensuring it is acceptable for practical uses<sup>[23,24]</sup>. The Si-O-Si backbone endows PDMS elastomers with intriguing properties, such as high flexibility, non-toxicity, non-flammability, thermal and electrical resistance, and low bulk density<sup>[25]</sup>. PDMS exhibits high transmittance and low absorption under UV irradiation and is suitable for desirable optical applications<sup>[26]</sup>. Because of its excellent contour accuracy of less than 10 nm, PDMS is widely exploited in micro- and nanotechnologies<sup>[22,27]</sup>. Solid PDMS is resistant to most aqueous reagents and alcohol solvents. However, organic solvents such as xylene can swell this elastomer<sup>[28]</sup>. Meanwhile, it is permeable to small, unreactive vapor and gas molecules such as water and oxygen<sup>[29,30]</sup>. Moreover, the surface of pristine PDMS presents low surface tension and energy and is hydrophobic. The wettability can be changed to hydrophilic temporarily by massively introducing hydroxyl groups with oxygen plasma treatment, yet it regains its hydrophobic property due to chain migration<sup>[31]</sup>. The PDMS surface can be easily modified via plasma oxygenation, adsorption of proteins, or conjugation of other functional chemical groups<sup>[32,33]</sup>. The high electronegativity can also be utilized to deposit oppositely charged electrolytes for hydrophilic modification and to realize extensive electrical applications<sup>[34]</sup>.

PDMS sponges, as one of the most important porous polymeric materials, involve quite a broad subject in relation to their chemical and physical structures, fabrication techniques, optimized properties, and desired applications<sup>[35]</sup>. To date, several excellent reviews on the development of porous polymeric materials have been published in the most recent years<sup>[25,36–43]</sup>. Readers can consult the comprehensive reviews to understand

structure design, synthesis strategies, correlated functions, and applications. However, PDMS sponges have been rarely mentioned in these existing reviews. Considering their outstanding properties and growing research interest, we believe that offering a focused review to summarize these newly emerged design strategies and applications is very important for developing elastic PDMS sponges.

On the other hand, the silica/polymer composite materials combine the rigidity and high thermal stability of the inorganic component with the organic polymer component's flexibility, ductility, and processability<sup>[44–51]</sup>. The silica nanoparticles have many interesting properties, including high mechanical strength, permeability, thermal and chemical stability, a relatively low refractive index, and a high surface area. Incorporating the nanoparticles into polymer matrices would considerably enhance their mechanical and thermal properties and improve their insulation properties. **Figure 2** shows the schematic illustration of silica/polymer composite fabrication. Therefore, polymer/silica composite materials can exhibit various unique properties, such as optical transparency, specific electrical and mechanical characteristics, thermal and weathering resistance, abrasion resistance, and impact resistance, for high-performance coating applications. Many other applications of silica/polymer composites include metal uptake<sup>[52–58]</sup>, sensors<sup>[59–62]</sup>, electronics and optical materials<sup>[63–65]</sup>, photoresist materials<sup>[66–68]</sup>, optical devices<sup>[69–71]</sup>, flame-retardant materials<sup>[72,73]</sup>, proton exchange membranes<sup>[74–78]</sup>, anticorrosion materials<sup>[79,80]</sup>, grouting materials<sup>[81,82]</sup>, oil adsorbents<sup>[83,84]</sup>, biomedical materials<sup>[85–88]</sup>, other coatings<sup>[89–91]</sup>, etc.

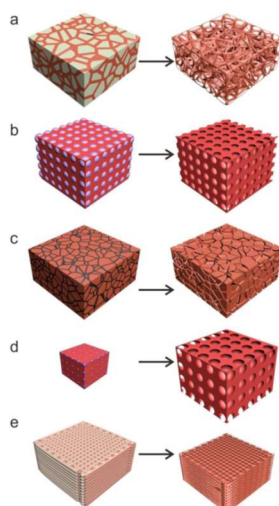


**Figure 2.** The schematic illustration of silica/polymer composites fabrication and its potential applications<sup>[92]</sup>. Reproduced with permission from Elsevier.

## 2. Oil-absorbents based on silicon-containing polymers

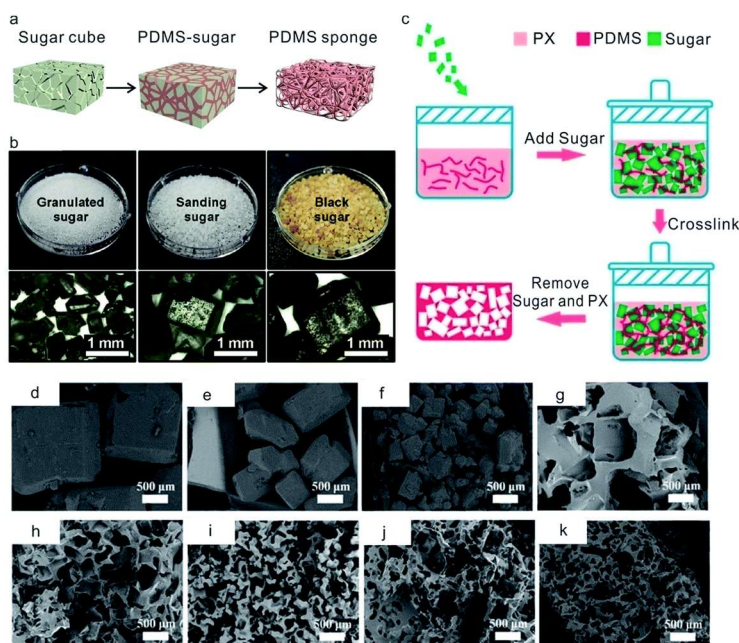
Therefore, it is evident that the PDMS is a widely studied system for oil-absorption applications among various silicon-containing polymers<sup>[12,93]</sup>. A straightforward and often utilized method for fabricating PDMS sponges is to use diverse solid templates, such as porogen, which can be selectively dissolved or removed to leave a PDMS skeleton with interconnected cavities. Based on the type of templates, this class of direct templating can be categorized into two main approaches using sacrificial templates, including salt crystals, sugar cubes, zinc oxide powders, nickel foam, and easily dissoluble polymer particles<sup>[94–99]</sup>. Among them all, the particulate leaching of salt or sugar cubes is the most prominent. A reason for its widespread use lies in the facile realization that no sophisticated laboratory equipment or hazardous solvent is required. In particular, water is utilized for leaching, and the leached salt or sugar particles/cubes can be retrieved afterward. Also, sugar leaching techniques are ready for scale-up production. For example, Wang et al. fabricated a bigger PDMS sponge by simply using sugar particles, which weathered a humidity of 85%<sup>[100]</sup>. The high humidity resulted in a fusion of the sugar particles, which ultimately yielded well-interconnected macroporous PDMS sponges.

Porous PDMS materials can be prepared using sugar, salt, self-assembled colloidal microspheres, emulsion droplets, and CO<sub>2</sub> gas generated by NaHCO<sub>3</sub> as templates, among which the sacrificial sugar template technique has been the most popular one because of its low cost and eco-friendliness<sup>[101–103]</sup>. **Figure 3** shows the schematic illustration of PDMS oil-absorbent preparation by template syntheses using various templates.



**Figure 3.** The schematic illustration of preparing PDMS oil absorbents using templated syntheses. **(a)** sugar cubes; **(b)** polystyrene beads; **(c)** Ni foam; **(d)** gas foaming; and **(e)** printed (3D-) templates<sup>[12]</sup>. Reproduced with permission from the Royal Society of Chemistry.

**Figure 4(a)** shows a typical sugar cube-leaching method for fabricating PDMS sponges<sup>[104]</sup>. Briefly, sugar cubes as the sacrificial template, are placed in a container ready for molding PDMS elastomers. Subsequently, a mixture of PDMS pre-polymer and curing agent (10:1 by weight) is poured into a pool to submerge the sugar cubes, followed by degassing in a vacuum chamber to assist the infiltration of liquid pre-polymer into the voids of the sugar cubes. Afterward, the PDMS-sugar cube is cured in an oven. Finally, the cured PDMS-sugar cubes are cut to reveal the sugar template, and the sugar portion is leached out by the water, resulting in 3D interconnected porous structures of PDMS sponges. Not limited to sugar cubes, Choi et al.<sup>[95]</sup> reported the fabrication of PDMS sponges with optimized performance in oil absorption by using sugar particles with different sizes, as shown in **Figure 4(b)**. Zhang et al.<sup>[13]</sup> reported a modified sugar leaching method by directly curing PDMS pre-polymer in a *p*-xylene (PX) solution in the presence of sugar particles, as shown in **Figure 4(c)**. As such, the as-made PDMS sponges have 3D interconnected pores and a swellable skeleton (**Figures 4(g)–(k)**).

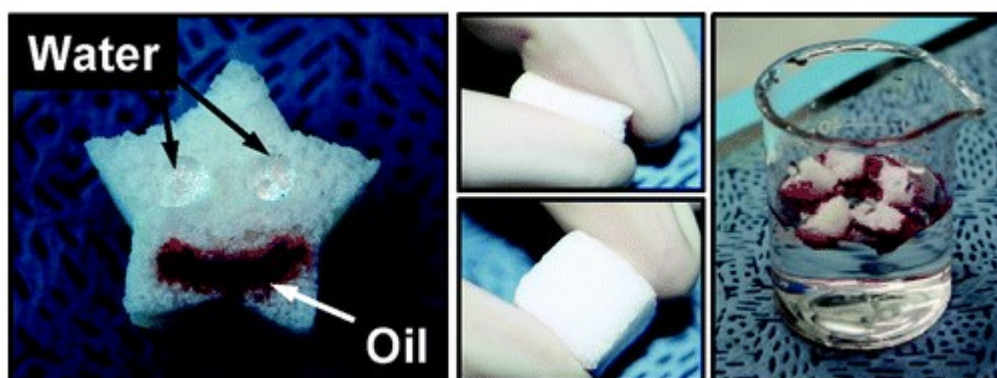


**Figure 4.** PDMS sponges fabricated by the sugar leaching method: **(a)** Schematic illustration of the fabrication process using a sugar cube as the template. **(b)** Photographs of sugar particles of different sizes. **(c)** A vacuum-free sugar-particle leaching method in a *p*-xylene (PX) solution, where the sugar and PX were removed after curing. **(d)–(k)** Scanning electron microscopy (SEM) images of sugar particles **(d)–(f)** and the as-made PDMS sponges **(g)–(k)**<sup>[12]</sup>. Reproduced with permission from the Royal Society of Chemistry.



PDMS sponges were also synthesized based on emulsion templates *via* the polymerization of the continuous phase of an emulsion, in which the emulsion droplets act as templates for pore formation<sup>[105–108]</sup>. The phase separation technique is promising for fabricating porous polymer membranes and 3D structures<sup>[109,110]</sup>. Jung et al.<sup>[105]</sup> systematically investigated the formation of pores during the fabrication process of a PDMS sponge and evaluated the performance of a homemade pressure sensor based on a PDMS sponge. Advances in the manufacture of additives have drawn much attention this century and have resulted in numerous improved approaches for 3D printing using PDMS<sup>[111–113]</sup>. However, the direct 3D printing of PDMS to form complex structures is still challenging, owing to the low elastic modulus of the liquid pre-polymer.

The oil and various organic solvent absorbencies of the PDMS sponge replicated from sugar templates were investigated in detail by Choi et al.<sup>[95]</sup>. The PDMS sponge was dipped into the organic solvents and oils for a few seconds. The oil-filled PDMS sponge could float on water without water penetration into the structure or the release of the oil; this was verified from the measured weight of the oil-soaked PDMS sponge, which changed by only 6% after 24 h (**Figure 5**). One important observation was that the absorbed oil was almost completely retained after the PDMS sponge was removed from the water, as evidenced by its weight remaining within 93% of the wet weight after 24 h. The PDMS sponge showed absorption capacities ranging from 400 wt% to 1100 wt% for various oils and organic solvents, with the maximum absorption capacity reaching up to 10 times its weight. The changes in absorption capacity depend on the density of the organic solvents and oils. Furthermore, the PDMS sponge also showed a high absorption capacity for nonpolar and polar organic solvents. For instance, the absorption capacity of 1,2-dichlorobenzene (a well-known toxic organic contaminant in water treatment) for the PDMS sponge based on the template with granulated and black sugar particles was approximately 1000 wt%. Because the PDMS materials show relatively excellent chemical inertness, the PDMS sponge may be used for nonpolar and polar organic solvents, with great potential for removing toxic organic contaminants and oil spills from water. Swelling of the PDMS sponge was occasionally observed along with the absorption of organic solvents because the organic solvents can diffuse into the PDMS material. Nevertheless, this swelling did not influence the sponge's absorption properties.

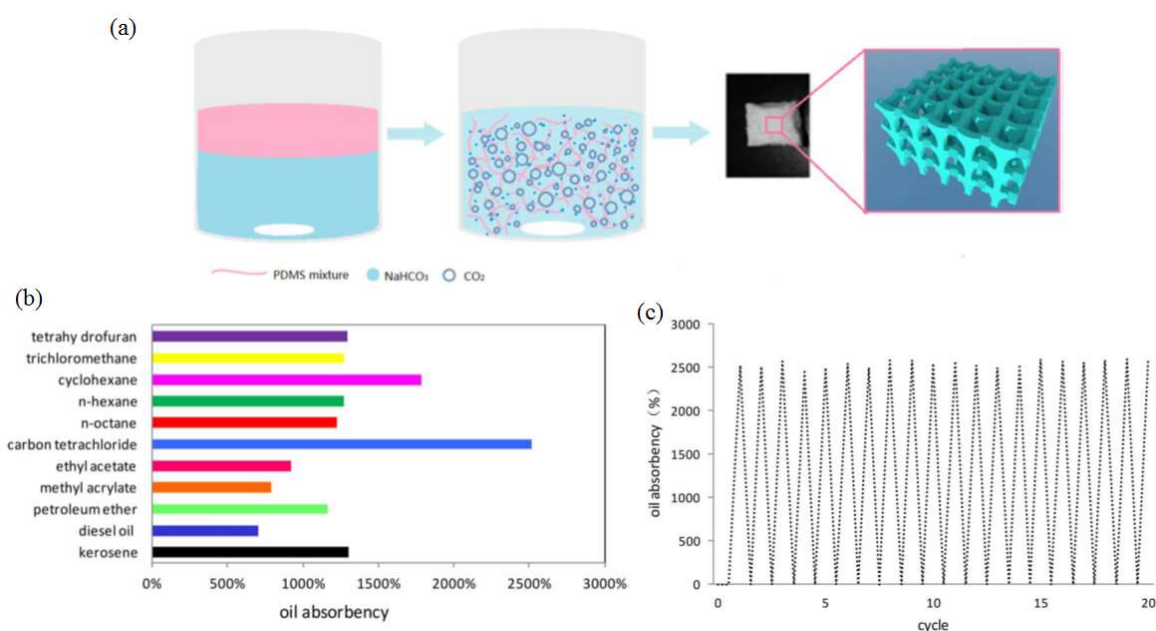


**Figure 5.** PDMS sponge prepared using the sugar template method for selective oil absorption from oil/water mixture<sup>[95]</sup>. Reproduced with permission from ACS.

The recyclability of the PDMS sponge and the recoverability of oils and organic solvents address critical requirements in practical oil clean-up applications. The absorbed oils and organic solvents in the PDMS sponge can be removed and reused by manually squeezing the PDMS sponge due to the springy nature of the PDMS material. As such, the fabricated PDMS sponge is desirable because it facilitates the recycling of oil-absorbent materials by allowing for the repeated capture and release of oils and organic solvents. To test the recyclability of the PDMS sponge as an absorbent material, the PDMS sponge can be squeezed and immediately immersed in an organic solvent (e.g., ethanol) 20 times after absorbing the transformer oil; the sponge weight was measured before and after drying. The reason for immersing the oil-absorbed PDMS sponge in ethanol was to ensure that any oil remaining after squeezing the oil-soaked PDMS sponge would be completely exuded into

the ethanol. The results show that the absorption capacity did not deteriorate, and the weight of a dry PDMS sponge did not notably change when the PDMS sponge was reused multiple times. When the PDMS sponge was immersed in water after the reusability test, it absorbed no water, indicating that hydrophobicity was not lost<sup>[95]</sup>.

Guo et al.<sup>[114]</sup> successfully synthesized a PDMS sponge by polymerizing the pre-polymer and a curing agent in a saturated  $\text{NaHCO}_3$  solution in the presence of carbon tetrachloride and n-octane (**Figure 6(a)**). Compared with the previous research, the PDMS sponges fabricated via simple and time-saving methods show excellent hydrophobicity/oleophilicity and thermal properties. The absorption capacities of the 3D interconnected porous PDMS sponges with swellable skeletons are 694%–2513% for various oils and solvents (**Figure 6(b)**). Due to their special wettability, the PDMS sponges can absorb oils and organic solvents from the oil-water mixture, and the whole separation process can be finished within 20 s. Furthermore, the oil absorption capacity of the PDMS sponge has no difference after 20 cycles and without weight loss (**Figure 6(c)**). Because of these excellent properties and simplicity of fabrication, it can be envisaged that the PDMS sponge will be a very promising material for handling oily wastewater.



**Figure 6.** (a) Preparation process of PDMS oil-absorbent; (b) oil and organic solvents absorptivity by the PDMS sponge; and (c) the recyclability of the absorbent. Reproduced with permission from John Wiley & Sons, Inc.<sup>[114]</sup>.

Compared with the fabrication of oil-absorbents, PDMS oil-absorbents have been easily prepared and have much more stability and practical applicability. Moreover, the preparation process involves the shape of sugar templates and the necessity of a vacuum operation, inducing penetration of PDMS pre-polymer into the templates. From the viewpoint of absorption mechanism, porous absorption materials store oils and organic solvents in pores, while gels retain them among the 3D cross-linked network of molecular chains or aggregates. Therefore, the higher the porosity, the larger the absorption capacity for porous absorbents<sup>[115]</sup>. However, considering the flexibility of the PDMS molecular chain, it is only possible to increase the porosity of PDMS with a limit. High porosity may result in a collapse of the porous structure<sup>[116]</sup>. These PDMS oil absorbents have interconnected pores and swellable skeletons, which can absorb and retain oils. The high oil absorption capacity of the PDMS sponge mainly ascribes to similar surface energies between oils (e.g., ethyl acetate,  $26.29 \text{ mNm}^{-1}$ ) and PDMS ( $20.4 \text{ mNm}^{-1}$ ). The sponge's surface strongly repels water due to a much higher surface tension ( $72 \text{ mNm}^{-1}$ ) than the PDMS. The capillary flow further improved the absorption capacity due to the interconnected pores of the PDMS sponge. After determining the weight of the water before and after separation, the separation efficiency is 97.9%. Compared with other sponges, PDMS sponges have excellent

advantages such as low cost, simple fabrication, good absorption capacity, and high separation efficiency<sup>[94,101,117]</sup>. The oil and organic solvent absorption capacities of various silicon-containing polymers have been summarized in **Table 1**. In addition to PDMS sponges, porous silicones have advantages when used for oil/water separation due to their high hydrophobicity, flexibility, thermal stability, and low costs<sup>[118–120]</sup>. Various porous silicones with high oil/water separation efficiency have been successfully synthesized<sup>[120,121]</sup>. For example, Moitra et al.<sup>[121]</sup> prepared porous hydrogen silsesquioxane monoliths by the sol–gel transition of trimethoxysilane, accompanied by a phase separation process.

**Table 1.** Summary of absorption capacities of various silicon-containing polymers.

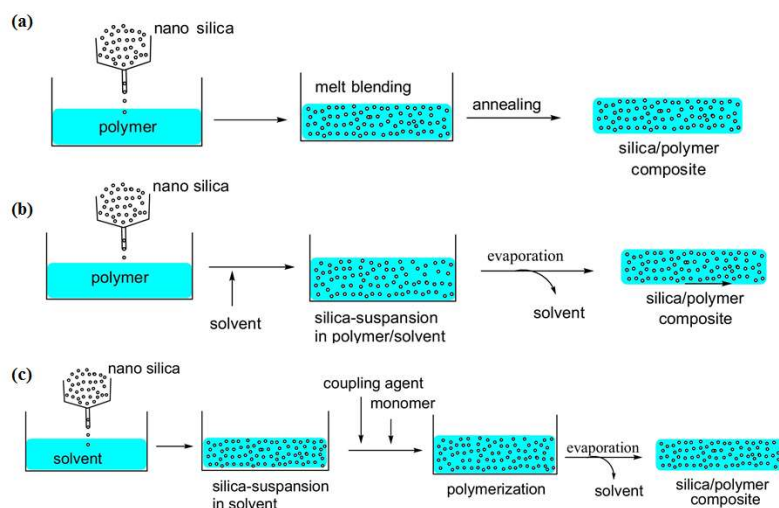
Absorbent material	Types of oil	Absorption capacity (g/g)	References
PDMS sponge	Various oils	4.72–20	[94]
PDMS sponge	Various oils	7.90–40	[122]
PDMS sponge	Various oils	6.9–25.2	[114]
Porous PDMS	CCl <sub>4</sub>	5.72	[117]
PU/CNT/PDMS sponge	n-Hexane	15	[123]
PU/PTFE/SiO <sub>2</sub> /polysiloxane sponge	Silicon oil	9.9	[124]

### 3. Polymer/silica composites as high-performance functional materials

In recent times, scientists have paid attention to a new type of coating: organic/inorganic composite coatings<sup>[125–132]</sup>. These coatings combine the flexibility and easy processing of polymers with the hardness of inorganic materials and have been successfully applied to various substrates<sup>[133–137]</sup>. These composite coatings are generally transparent, show good adhesion, and enhance a polymeric substrate's scratch and abrasion resistance<sup>[136,138–145]</sup>. The reinforcement of acrylates by surface-modified nanosilica led to acrylate nanocomposite coatings with improved scratch and abrasion resistance<sup>[146]</sup>. These coatings can be used on substrates such as polymer films, paper, metal, wood, and engineered wood. In addition, compared with nanocomposite materials, much better abrasion resistance was obtained for coatings containing both silica nanoparticles and corundum microparticles. These nano- and micro-composites are recommended as clear coats for parquet and flooring applications. It was shown that polymer/silica nanocomposites can be obtained in various structures and compositions by using miniemulsion polymerization<sup>[147–150]</sup>. The resulting structures are possibly interesting for generating waterborne coatings, which show the polymer's ability for spontaneous film formation in combination with the high mechanical scratch resistance provided by the inorganic nanoparticles.

The simplest method for preparing polymer/silica composites is directly mixing the silica and the polymer. The mixing can generally be carried out by melt blending, solution blending, and in situ polymerization (**Figure 7**). In such mixing processes, the primary key point is the effective dispersion of the silica nanoparticles into the polymer matrix because they generally tend to agglomerate. Melt blending is most commonly utilized because of its effective processability<sup>[151]</sup>. This method has some advantages, such as being simple, cost-effective, easy to process, and widely applicable. Using a twin screw extruder and injection molding machine, the mechanical property of polymer composites loaded with nanoparticles could be better than that of a conventional compounding route in which the nanoparticles were pre-grafted by some polymers through irradiation. Melt blending for the composite of silica and the polymers is generally carried out above the polymer's glass-transition temperature ( $T_g$ ). This mixing route is more efficient and operable than mechanically simple mixing at room temperature. This method is a polymer-inorganic powder blending process using a solvent in which the polymer is soluble. This processing is widely used for common composite preparation. In the dispersion of silica fillers into a polymer matrix, solution blending is much more effective

than melt blending<sup>[152]</sup>. This method can make nanofillers dispersible effectively into the polymer chains. With solvent removal, a well-dispersed silica/polymer composite can be obtained. However, there are drawbacks to the use of environmentally unfriendly organic solvents, including the selection of an appropriate solvent and the removal process of the solvent after composition, even though it can overcome the limitation of melt blending, which is not feasible due to severe shear heating and the formation of particle aggregates<sup>[45]</sup>. Polymer/mesoporous silica composites were commonly carried out using solution blending<sup>[153–155]</sup>. In the *in-situ* polymerization method, inorganic fillers are suspended in the liquid phase of the monomer, and then the polymerization can occur around and between the filler particles. The reaction can be initiated either by incorporating an initiator curing agent or by thermal activation and UV irradiation (enzymatic initiation).



**Figure 7.** (a) Preparation processes of silica/polymer composites; (a) melt blending; (b) solution blending; and (c) *in-situ* polymerization method. Reproduced with permission from Elsevier<sup>[92]</sup>.

Silica particles are used in functional coatings mainly in two forms: colloidal and fumed silica. Fumed silica, produced via flame hydrolysis, has been frequently used due to its significantly lower cost than colloidal silica. However, when dispersed, fumed silica tends to aggregate irreversibly. The preparation of colloidal silica particles usually follows the method introduced by Stöber, which consists of the hydrolysis and condensation of a silica precursor, such as tetraethyl orthosilicate (TEOS), in primary conditions<sup>[156]</sup>. Using this method, the particle size distribution is relatively narrow and tunable to different diameters, ranging from 20 nm to 1  $\mu\text{m}$ . The films' silica content must be optimized to increase tensile strength and impact resistance simultaneously without decreasing flexural properties. A silica content of 10 wt% is usually found to give the best film properties<sup>[157,158]</sup>.

### 3.1. Superhydrophobic coatings

Superhydrophobic surfaces have received considerable attention from fundamental research and practical applications<sup>[159]</sup>. Superhydrophobicity is defined as a water contact angle larger than  $150^\circ$  and contact angle hysteresis less than  $10^\circ$ , which is caused by the combined effect of both a hierarchical micro/nano-structure on the surface and a low surface energy<sup>[160–162]</sup>. Due to their water-repellent property, superhydrophobic surfaces have several emerging applications in a large number of fields, such as microfluidics<sup>[163]</sup>, self-cleaning-fabrics<sup>[164]</sup>, anti-icing materials<sup>[165,166]</sup>, anti-fogging surfaces, anti-corrosive industrial parts and even surface drag reduction to reduce energy consumption in transport systems<sup>[167,168]</sup>. As an example, Bravo et al.<sup>[169]</sup> demonstrated a layer-by-layer processing scheme that can be utilized to create transparent superhydrophobic films from  $\text{SiO}_2$  nanoparticles of various sizes. By controlling the placement and level of aggregation of differently sized  $\text{SiO}_2$  nanoparticles within the resultant multilayer thin film, it is possible to optimize the level of surface roughness to achieve superhydrophobic behavior with limited light scattering. Transparent

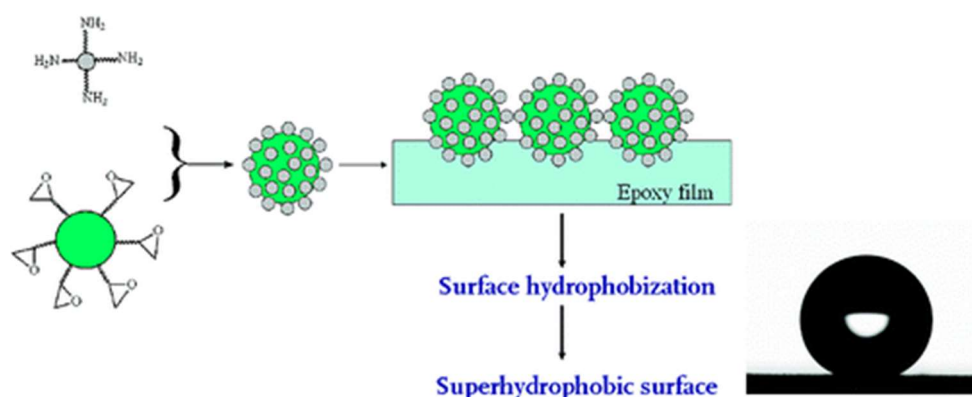


superhydrophobic films were created by sequentially adsorbing silica nanoparticles and poly(allylamine hydrochloride). The final assembly was rendered superhydrophobic with a silane treatment, as shown in **Figure 8**.



**Figure 8.** Photograph of the glass slide coated with superhydrophobic multilayer based on silica nanoparticles and poly(allylamine hydrochloride). Reproduced with permission from the American Chemical Society<sup>[169]</sup>.

Ming et al.<sup>[170]</sup> reported a robust procedure for preparing superhydrophobic films based on silica/polymer composites with an advancing contact angle for water of about  $165^\circ$  and the roll-off angle of a  $10\text{-}\mu\text{L}$  water droplet is  $3 \pm 1^\circ$ . Dual-size surface roughness mimics the surface topology of self-cleaning plant leaves originating from well-defined silica-based raspberry-like particles covalently bonded to an epoxy-based polymer matrix, as shown in **Figure 9**. The roughened surface is chemically modified with a layer of poly(dimethylsiloxane) (PDMS). The robustness and simplicity of this procedure would make widespread applications of so-prepared superhydrophobic films possible.



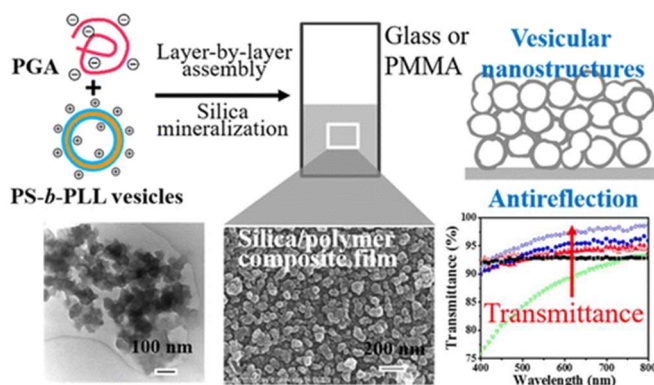
**Figure 9.** The schematic illustration is a superhydrophobic surface based on a silica/epoxy composite. Reproduced with permission from the American Chemical Society<sup>[170]</sup>.

Superhydrophobic thermoplastic polyurethane (TPU) films based on octadecanamide (ODAA)-directed assembly of nanosilica/TPU/ODAA hybrids with a well-defined sheet-like microstructure were achieved by Yang et al.<sup>[171]</sup>. Such superhydrophobic surfaces showed improved mechanical robustness, and the procedures exhibited were facile and versatile. Tang et al.<sup>[172]</sup> reported superhydrophobic coatings based on two polymers, epoxy and acrylate copolymers, combined with silica nanoparticles.

### 3.2. Anti-reflective coatings

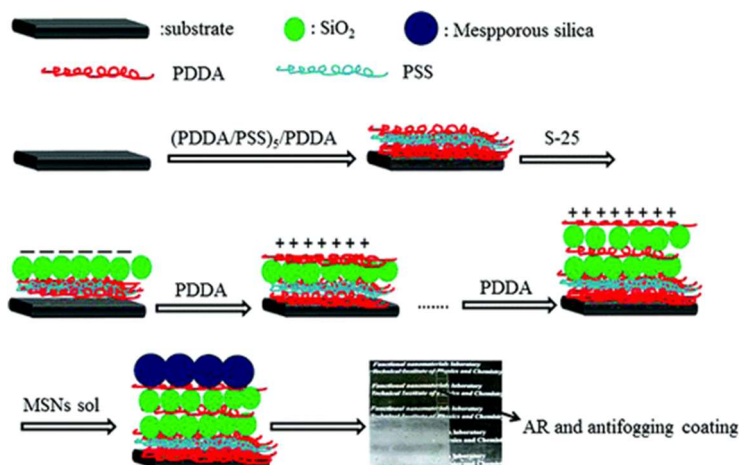
Anti-reflective films based on silica/polymer composites have been widely studied<sup>[173–177]</sup>. Lin et al.<sup>[178]</sup> reported anti-reflective silica/polymer composite coatings on glass and poly(methyl methacrylate) (PMMA) substrates. The coatings were prepared by silica mineralization of layer-by-layer (LbL) assembled films composed of polystyrene-block-poly(l-lysine)/poly(l-glutamic acid) (PS-b-PLL/PGA) complex vesicles

without any post-treatment. PS-*b*-PLL AB and A<sub>2</sub>B block copolymers with appropriate block ratios can self-assemble to form vesicles, which can be deposited onto substrates without dissociation. Silica deposition, specifically onto the complex vesicles in the multilayer films through amine-catalyzed polycondensation, results in continuous, intact composite coatings comprising vesicular nanostructures, which provides an additional parameter for tuning their optical properties. The film thickness and porosity are mainly dictated by the bilayer number and the degree of deformation or fission of vesicles upon complexation and mineralization, depending on polymer composition. The coated PMMA substrate with maximum transmission over 98% can be achieved in the optimized wavelength region (**Figure 10**). The AR composite films were mechanically stable enough to withstand the wipe and adhesion tests due to the preparation of continuous, intact films.



**Figure 10.** The anti-reflective coatings are based on silica/polymer composite film. Reproduced with permission from the American Chemical Society<sup>[178]</sup>.

Xu et al.<sup>[179]</sup> reported anti-fogging and anti-reflection coatings based on silica nanoparticles and mesoporous silica nanoparticles onto glass and PMMA substrates via LbL assembly without post-treatments (**Figure 11**). A maximum transmittance of 98.5% in the visible spectral range was achieved for the coating deposited on slide glass. The maximum transmittance even reached as high as 99.3% by applying a coating to the PMMA substrate. The high porosity of mesoporous nanoparticles and loose stacking of solid and mesoporous nanoparticles are considered to contribute significantly to the enhancement of light.



**Figure 11.** The anti-fogging and anti-reflective coatings were based on silica/polymer composite films fabricated by the LbL method. PSS-Sodium poly(4-styrene sulfonate); PDDA-poly(diallyldimethyl ammonium) chloride. Reproduced with permission from the American Chemical Society<sup>[179]</sup>.

### 3.3. Anti-corrosive and protective coatings

Among a large number of reported organic-inorganic composite systems in which polymers such as epoxy, polyimide, acrylic, and polyethylenimine phases are combined with inorganic oxides such as silica, one important class is the PMMA-silica system. PMMA-silica nanocomposites have recently received considerable

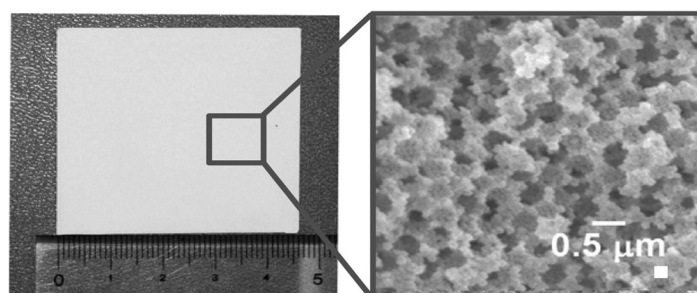
attention because of their ability to protect a wide variety of metal surfaces, such as steels, stainless steels, aluminum alloys, and magnesium alloys, in an efficient and environmentally compliant manner<sup>[180–182]</sup>. These alloys are crucial for critical aerospace, automotive, and offshore industries. In the case of PMMA-silica hybrid nanocomposites, this corrosion protection is a consequence of the covalent bonding between PMMA and silica nodes through the coupling agent 3-(trimethoxysilyl)propyl methacrylate (MPTS), formed by three methoxy-silane groups linked by a nonhydrolysable Si-C bond to a methacrylate tail. This bonding mechanism produces a class II hybrid with a nanostructure of dense silica cross-link nodes bridged by short polymeric chains; consequently, the closely packed nanostructure acts as an efficient corrosion barrier against the uptake of aggressive agents.

## 4. Other applications

### 4.1. Proton exchange membranes

The proton exchange membrane (PEM) is one of the significant components in solid-type fuel cells, such as the proton exchange membrane fuel cell (PEMFC) and the direct methanol fuel cell (DMFC). Up to now, many research groups have reported the fabrication of polymer/silica nanocomposites as PEM<sup>[76,183–190]</sup>. Sulfonated poly(phthalazinone ether ketone) (sPPEK) with a degree of sulfonation of 1.23 was mixed with silica nanoparticles to form hybrid materials for use as PEMs<sup>[191,192]</sup>. The hybrid membranes exhibited improved swelling behavior, thermal stability, and mechanical properties. The methanol crossover behavior of the membrane was also depressed, such that these membranes were suitable for a high methanol concentration in the feed in a cell test. Sulfonated P(St-co-MA)-PEG/silica nanocomposite polyelectrolyte membranes were prepared with varied silica content using PEG of different molecular weights to have a fine control over spacing between silica domains, up to a few nanometers, by a chemically bound interior polymer chain<sup>[193]</sup>. Moreover, this system showed clear improvement over the Nafion membrane, as seen by selectivity parameter values due to low methanol permeability at 30 and 70 °C. In contrast, Nafion showed almost the same selectivity parameter values at both temperatures. The relatively high selectivity parameter values of these membranes at 70 °C indicated an excellent advantage for the composite over Nafion 117 membranes for targeting higher temperature applications.

Kanamura et al.<sup>[194]</sup> reported a new proton exchange membrane based on macroporous silica and a proton-conducting gel polymer electrolyte (**Figure 12**). 3D-uniformly ordered macroporous silica was synthesized using a colloidal template method with monodisperse polystyrene beads. A gel polymer, 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS), was injected into all the pores to prepare a proton-conducting membrane composite of macroporous silica and the gel polymer. The prepared membrane exhibited high proton conductivity and low methanol permeation. An H<sub>2</sub>-O<sub>2</sub> fuel cell was constructed to test the composite membrane. The electrochemical performance obtained for the fuel cell with the composite membrane was similar to that for a fuel cell with a Nafion 117 membrane. From this result, it can be concluded that ceramic and polymer composite membranes can be applied to fuel cells working at low temperatures.



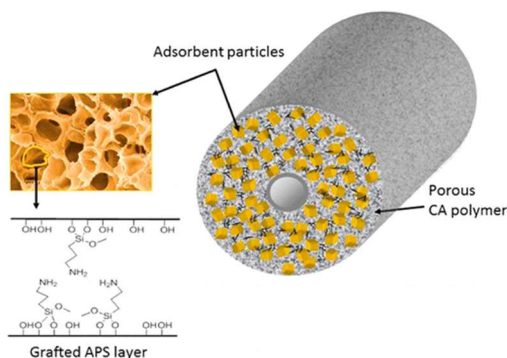
**Figure 12.** A new type of proton exchange membrane based on macroporous silica and a proton-conducting gel polymer electrolyte<sup>[194]</sup>. Reproduced with permission from the American Chemical Society.

## 4.2. Encapsulation for organic light-emitting devices

Direct encapsulation of organic light-emitting devices (OLEDs) is realized using highly transparent, photocurable *co*-polyacrylate/silica nanocomposite resin<sup>[195]</sup>. The feasibility of such a resin for OLED encapsulation was evaluated by physical/electrical property analysis of resins and driving voltage/luminance/lifetime measurement of OLEDs. Electrical property analysis revealed a higher electrical insulation of photocured nanocomposite resin film at  $3.20 \times 10^{12} \Omega$  in comparison with that of oligomer film at  $1.18 \times 10^{12} \Omega$  at 6.15 V to drive the bare OLED. This resulted in a lower leakage current, and the device driving voltage was efficiently reduced so that the nanocomposite-encapsulated OLED could be operated at a lower driving voltage of 6.09 V rather than 6.77 V for the oligomer-encapsulated OLED at the current density of 20 mA/cm<sup>2</sup>. Luminance measurements revealed a luminance difference of less than 1.0% between OLEDs encapsulated by various types of resins, which indicated that the photopolymerization had a minimal effect on the light-emitting properties of OLEDs. Lifetime measurement of OLEDs found that  $t_{80}$ , the time span for the normalized luminance of the device, drops to 80% for nanocomposites-encapsulated OLED, which is 350.17 h in contrast to 16.83 h for bare OLED and 178.17 h for oligomer-encapsulated OLED. This demonstrated that nanocomposite resin with optimum properties was feasible for OLED packaging, and a compact device structure could be achieved via direct encapsulation.

## 4.3. CO<sub>2</sub> capture

Recently, silica/polymer composites have been exploited for CO<sub>2</sub>-capturing applications<sup>[196–199]</sup>. Rezaei et al.<sup>[200]</sup> reported amine/silica/polymer composite hollow fiber adsorbents, which are produced using a novel reactive post-spinning infusion technique, and the fibers are shown to capture CO<sub>2</sub> from simulated flue gas (**Figure 13**). The post-spinning infusion technique allows for functionalizing polymer/ silica hollow fibers with different types of amines during the solvent exchange step after fiber spinning. The post-spinning infusion of 3-aminopropyltrimethoxysilane (APS) into mesoporous silica/cellulose acetate hollow fibers is demonstrated here, and the materials are compared with hollow fibers infused with poly(ethyleneimine) (PEI). This approach results in silica/polymer composite fibers with good amine distribution and accessibility and adequate porosity retained within the fibers to facilitate rapid mass transfer and adsorption kinetics. In contrast, fibers that are spun with presynthesized, amine-loaded mesoporous silica powders show negligible CO<sub>2</sub> uptake and low amine loadings because of the loss of amines from the silica materials during the fiber spinning process. Aminosilica powders are shown to be more hydrophilic than the corresponding amine-containing composite hollow fibers, the bare polymer, as silica supports. The PEI-infused and APS-infused fibers demonstrate reduced CO<sub>2</sub> adsorption upon elevating the temperature from 35 to 80 °C, per thermodynamics. In contrast, PEI-infused powders show increased CO<sub>2</sub> uptake over that temperature range because of competing diffusional and thermodynamic effects. The results indicate the post-spinning infusion method provides a new platform for synthesizing composite polymer/silica/amine fibers that may facilitate the ultimate scale-up of practical fiber adsorbents for flue gas CO<sub>2</sub> capture applications.



**Figure 13.** Amine/silica/polymer composite hollow fiber adsorbents<sup>[200]</sup>. Reproduced with permission from the American Chemical Society.



## 4.4. Sensors

Silica/polymer composites have been widely studied for various sensing applications<sup>[201–205]</sup>. An approach for preparing polydiacetylene/silica nanocomposites for use as a chemosensor was reported<sup>[62]</sup>. The disordered 10,12-pentacosadiynoic acid (PCDA) aggregates could be absorbed on the surfaces of silica nanoparticles in an aqueous solution. The disordered PCDA molecules in aggregates were turned into an ordered arrangement with the help of a silica nanoparticle template. After irradiation with UV light, polydiacetylene/silica nanocomposites took on a blue color. Various environmental perturbations, such as temperature, pH, and amphiphilic molecules, could result in a colorimetric change of the polydiacetylene/silica nanocomposites from the blue to the red phase. The material may find some interesting potential applications as a new chemosensor.

## 4.5. Metal uptake

The nanocomposites of electroactive polymers PANI or PPy with ultrafine SiO<sub>2</sub> particles have potential commercial applications for metal uptake based on the fact that they possess a surface area substantially higher than that estimated from the particle size and hence can aid the process of metal uptake. The use of electroactive polymer/SiO<sub>2</sub> nanocomposites to uptake gold and palladium from AuCl<sub>3</sub> and PdCl<sub>2</sub> in acid solutions, respectively, was investigated<sup>[53]</sup>. In the case of gold uptake, the reaction rate increased with temperature from 0 to 60 °C. The accumulation of elemental gold on the nanocomposites increased the diameter and decreased the surface area. The surface Au/N ratio, as determined using XPS, was highly dependent on the rate of reactions, even for the same amount of gold uptake. The uptake of palladium from PdCl<sub>2</sub> was much more challenging to accomplish. High uptake rates could only be achieved with the electroactive polymers reduced to their lowest oxidation state. Unlike the case of gold uptake, the palladium on the microparticles did not exist in the elemental form but as a Pd(II) compound.

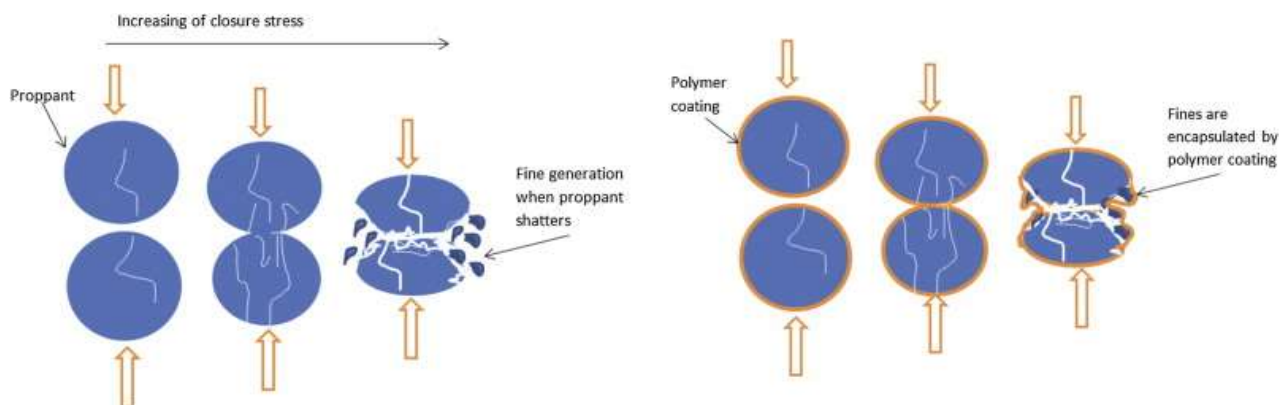
# 5. Functional materials based on sand

## 5.1. Proppants

A proppant can be defined as a solid material, usually sand or modified sand, designed to keep the induced hydraulic fracture open to extract gaseous products from the fracture. The so-called frac sand is easily friable and creates fines when over-stressed; modified sand with polymers or resins was developed to enhance the conductivity of frac sand. The main advantage of using resin to coat the proppant is that the resin coating can trap pieces of broken grain within the coating, thereby preventing proppant flow back to the wellbore (**Figure 14**). They could also connect individual proppant grains to avoid the proppant flowing back. In this way, they are commonly used in the tail-in-fracturing process. The details of different types of proppants and their functions, developed and used in the oil and gas industries, can be found elsewhere<sup>[206,207]</sup>. The resin-coated proppants can also prevent sand production in areas of soft formation where sand control is needed<sup>[208–211]</sup>. The resin coat can be pre-cured or curable. In general, uncured resin systems have poor mechanical properties. However, good properties are obtained by reacting the linear resin with suitable curatives to form 3D-cross-linked thermoset structures. This process is commonly referred to as curing. Pre-cured, resin-coated sand is processed by applying the resin to the sand. No further curing will take place downhole. For curable resin-coated proppant, the well is shut in after fracturing to allow curing—the curing process results in a consolidated proppant bed with a cured resin coating surrounding each proppant grain. The performance of the proppant depends on the properties of the cured resin material. The chemical cross-links that form during the resin cure do not allow the cured material to melt or flow when re-heated. However, cured/cross-linked resins undergo a very slight softening at elevated temperatures at a point of  $T_g$ . When the temperature is above the  $T_g$ , the mobility of the polymer chains increases significantly, and the cured resin changes from a rigid/glassy state to

a rubbery/compliant state. In this case, the resin system becomes soft, decreasing strength. Therefore, the  $T_g$  has been used as a valuable parameter to determine the upper-performance limit of the resin.

The most commonly used resins used to coat proppants are epoxy resins, furan, polyesters, vinyl esters, and polyurethane<sup>[207]</sup>. Epoxy resin is the primary polymer type used for proppant coating because it has excellent mechanical strength, heat resistance, and chemical resistance.

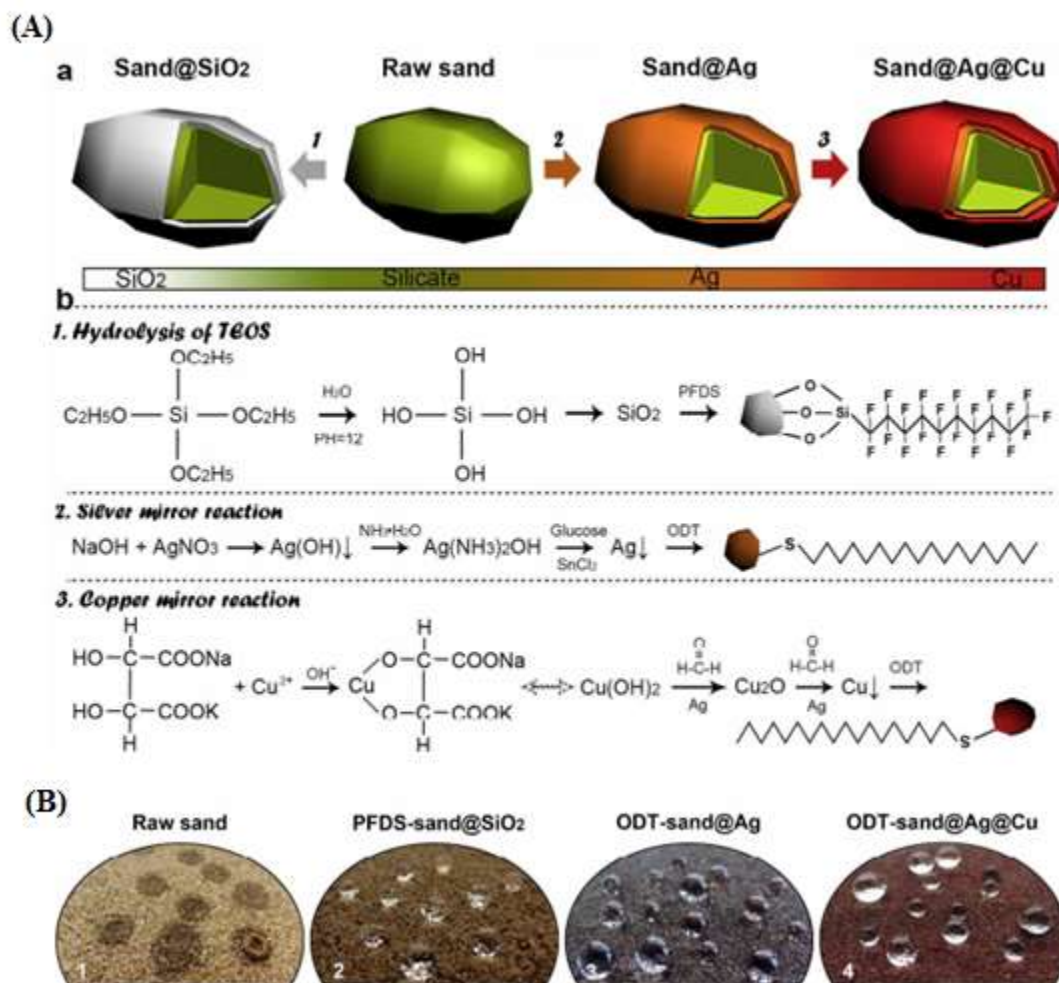


**Figure 14.** The schematic illustration of fine production in unmodified and modified (polymer coated) proppants under closure stress<sup>[207,212]</sup>. Reproduced with permission from Elsevier.

## 5.2. Superhydrophobic sand

“Superhydrophobic sand” has recently been studied for various potential applications<sup>[213–215]</sup>. Superhydrophobic sand has been proposed to realize the storage and transportation of the surface water of sandy soils, especially in deserts<sup>[214,216]</sup>. Sand dunes are an abundant natural resource in the desert, are characterized by a low water storage capacity, and suffer from a temporary water shortage, mainly when cultivated under arid conditions. Sand consists of hydrophilic silica, which attracts water and facilitates its flow to the ground, thus adversely affecting the plantation, even under expensive irrigation systems such as sprinkling or trickling. Previously, hydrogels were used to increase the water-holding capacity of sandy soils<sup>[217–219]</sup>. Recently, it has been proposed that applying nanomaterials to the sand surface can combat desertification and encourage plant growth in arid climates. Moreover, superhydrophobic sand prevents the diffusion of underground salt, affecting plants' growth. Applying the superhydrophobic properties of certain nanomaterials to mitigating environmental and resource issues is a promising research topic of global significance. The development of low-cost, environmentally friendly, superhydrophobic, thermally stable, and promising adhesion nanomaterials is a challenge for desert greening.

Chen et al.<sup>[220]</sup> reported the preparation of three kinds of hydrophobic sands with different surface structures and wettability by cladding inorganic materials of nonmetal ( $\text{SiO}_2$ ) and metal (Ag and Cu) on sand surfaces and then modifying them with low-surface-energy chemicals (**Figure 15**). Combining the superhydrophobicity with the desert sand, superhydrophobic sand (PFDS-sand@ $\text{SiO}_2$ ) is shown to have excellent water repellency, meaning that the water can stably stay and flow on such a sand surface without any wetting or permeation, and has such excellent water-holding capacity that a sand layer with a 2 cm thickness can sustain a water column height of 35 cm. Significantly, such superhydrophobic sand, PFDS-sand@ $\text{SiO}_2$ , exhibits exceptionally high thermal stability up to 400 °C when used for water storage. This result is unprecedented and enough to face the high-temperature conditions of the desert and some others.



**Figure 15.** (A) The preparation of superhydrophobic sands. (a) Schematic illustration of the surface modifications of the sand; (b) the chemical reactions of the surface modifications of the sand. (B) The wettability and water repellency of the prepared superhydrophobic sands<sup>[220]</sup>. Reproduced with permission from the Royal Society of Chemistry.

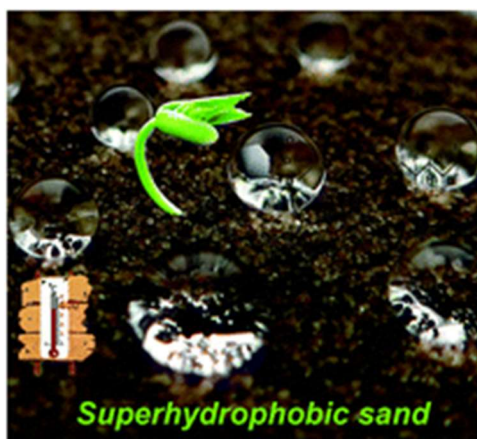
### 5.3. Nanosand-based functional materials

Nanosand derived from highly abundant and low-cost sand through nanomilling has attracted the great interest of researchers recently, as it finds various potential applications, including as a critical component in nanofluids<sup>[221,222]</sup>, water filtration membranes<sup>[223]</sup>, as a nanofiller<sup>[224–229]</sup>, and polypropylene-based composites<sup>[230]</sup>. In addition, nanostructured sand is found to be a potential candidate for oil recovery in produced water treatment and pollutant absorption<sup>[231]</sup>. Manikandan et al.<sup>[222]</sup> reported the stable dispersions of nanosand with propylene glycol (P.G.), an excellent coolant system for heat transfer management in various manufacturing units. Similarly, they have also studied the sub-micron-sized sand dispersion in water as a nanofluid<sup>[221]</sup>. The stable dispersion of nanoparticles in a liquid, stabilized either by electrostatic or steric interaction, has provided a practical method of improving the thermal conductivity of conventional coolant through particle addition. Such stable dispersions are referred to as nanofluids. Nanosand-water or nanosand-propylene glycol dispersions have a higher thermal conductivity ratio than silica-water nanofluids reported in the literature. Stable nanosand-PG nanofluids were formulated by dispersing nanosands of 20–25 nm particle size produced by stirred bead milling in P.G. through extended probe ultrasonication. The viscosity of nanosand-PG nanofluids decreases with the concentration of nanosand and temperature. In the well-dispersed nanosand-PG, the interactions between nanoparticles and P.G. through the nanoparticle surface led to disturbance of the hydrogen bonding network of P.G. This results in a reduction in the viscosity of the nanofluid in comparison to pure P.G. For instance, the viscosity of 2 vol% nanosand-PG nanofluid is found to be 46% lower than that of pure P.G. at 29 °C.

Nanosand has been introduced into epoxy resin for its good toughness and mechanical strength. The nanosand exhibited good compatibility with the epoxy matrix, and the dispersion and concentration of the nanosand determined the enhancement. Both flexural strength and fracture toughness have been greatly improved by incorporating 3 wt% nanosand. The addition of nanosand to epoxy changes the curing reaction and the  $T_g$ . The nanosand has been used without any further surface treatment, resulting in a lot of hydroxyl groups (-OH) groups and, hence, better chemical interaction with the epoxy resin and better properties of the composites.

## 6. Summary and outlook

Recently, more research has been published on silica or sand/polymer composites aimed at various potential applications. The composites' current status and recent application developments have been summarized. The different preparation methods of silica and polymer have been extensively investigated. However, the properties of polymer/silica nanocomposites are generally superior to those of pure polymer matrixes and polymer microcomposites. Multi-functional coatings based on silica/polymer composite materials combine the flexural properties and processability of the polymer with a varied array of properties that the inorganic components can impart. Silica nanoparticles are among the most commonly used for composite coating applications. These not only provide increased tensile strength, scratch resistance, and impact resistance but are also extremely versatile vehicles to incorporate other components, which, in turn, open the possibility of obtaining coatings with a wide range of new properties. Most of the properties and potential functionalities of these silica/polymer composites are dependent on a homogeneous distribution of the organic and inorganic components. For example, functionalized polymer nanoparticles can serve as a template to prepare silica composite films with a hierarchical structure to obtain superhydrophobic and amphiphobic coatings for self-cleaning surfaces. The silica nanoparticles are incorporated in a polymer matrix in most of the of the described applications. Due to mixability issues of the silica with most polymers, this requires the surface functionalization of the silica to achieve good compatibility between the two components and, thus, avoid the aggregation of the silica particles in the final material. Using composite nanoparticles with a silica core and a polymer shell can obtain perfectly homogeneous particle distributions and further control the spacing between silica particles in the final material, which might be necessary for many applications<sup>[16,43,232–239]</sup>. The facile oil-water separation, absorption, and CO<sub>2</sub> capture by the silica/polymer composites are promising in overcoming recent environmental issues. Very interestingly, considerable efforts have been made to realize agriculture in deserts through superhydrophobic sand (**Figure 16**). All these manifestations imply the significant potential of such superhydrophobic sand in its application to desert water storage and transportation. The facile and economical preparation methods of the superhydrophobic sand would be a big hope in achieving the most challenging water storage and transport in Middle Eastern countries.



**Figure 16.** A photograph depicts the realization of agriculture through superhydrophobic sand in deserts<sup>[220]</sup>. Reproduced with permission from the Royal Society of Chemistry.



## Acknowledgments

This study is part of research project agreement no. AFU-01-2017 in collaboration with EXPEC Advanced Research Centre, Saudi Aramco. The authors gratefully acknowledge the continued support from Alfaisal University and its Office of Research.

## Conflict of interest

All authors declare no conflict of interest.

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