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A Review: Synthesis, Recent Application and Future Challenge of SiO_2 Nanoparticles in Membrane Technology

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Abstract

Membrane processes are well-established methods in real water treatment, renowned for their compact size, exceptional treatment effectiveness, and substantial water permeation capacity. The emergence of nanomaterials has ushered in thrilling prospects and progress in the membrane technology. Silica nanoparticles (NPs SiO_2), a high-purity amorphous silica powder, is a white fluffy material characterized by its small particle size, and it brings numerous benefits due to its distinctive attributes. With a large specific surface area, strong surface adsorption, considerable surface energy, high chemical purity, and excellent dispersion, it holds significant value in diverse areas like medicine, physics, chemistry, and biology. NPs-SiO_2 can be categorized into hydrophilic and hydrophobic types, which offers difference application in the membrane technology. In this review, NPs-SiO_2 contained membranes are specifically discussed. The main goal is to provide critical analysis on recent application of SiO_2 in membrane technology. Apart from the introduction and conclusion, this review has three consecutive parts including The synthesis methods of NPs-SiO_2 contained membrane including size of the NPs-SiO_2 that is mostly used in membranes, the application of the hydrophobic and hydrophilic NPs-SiO_2 in the membrane technology, and the future challenge of NPs-SiO_2 used in membrane technology.

Keywords: hydrophilic NPs-SiO_2, hydrophobic NPs-SiO_2, membrane technology

1 Introduction

Membrane processes are techniques widely known in the field of water treatment, which has a small footprint, high treatment efficiency, and a large amount of permeated water capacity [1]. Membranes are now ubiquitous to all industries. They are prevalent in water and wastewater treatment, food and beverage processing, pharmaceutical production, industrial gas production, commodity and specialty chemicals, and barrier materials [2].

Over the past few decades, the membranes market has experienced steady growth. Approximately 60% of the membrane area and sales are attributed to various microfilters, while both ultrafiltration and reverse osmosis account for nearly 17% each [3]. The remaining 8% is divided among other membrane technologies, with pervaporation occupying around 1.5% of that share. In terms of membrane materials, polymers dominate, constituting approximately three-quarters of the commercially available membranes, while the rest are composed of ceramic, metallic, and other inorganic materials. [4].

Conventional polymeric membranes are extensively utilized primarily because of their uncomplicated pore-forming process, excellent flexibility, easy installation, and comparatively lower cost compared to inorganic membranes (5). However, inorganic membranes have emerged as strong commercial contenders for various reasons, including their superior chemical resistance, enhanced mechanical strength, improved conversion rates, and superior selectivity when compared to polymeric membranes [6].

However, these conventional membranes are not without their limitations. One of these limitations involves a trade-off between permeability and selectivity. Improving the permeability of a material often comes at the expense of its selectivity, and vice versa [7]. Additionally, there are significant drawbacks related to energy consumption, fouling, and overall operational costs, which hinder their widespread commercial use. Membrane processes driven by pressure, for instance, suffer from issues like fouling that limit their potential for commercialization as they are prone to deterioration in permeate quality and early membrane replacement [8]. Conversely, membrane processes not driven by pressure face constraints such as reduced permeation and fouling susceptibility [9]. As a result of these limitations, researchers have shifted their focus towards exploring advanced membrane-nanomaterial composites in search of superior alternatives.

Nano-SiO_2, a high-purity amorphous silica powder, appears as a white fluffy substance with small particle size, offering several advantages. Its unique properties, including a large specific surface area, strong surface adsorption, significant surface energy, high chemical purity [10], and excellent dispersion, make it highly valuable in various fields such as medicine, physics, chemistry, and biology [11]. Depending on its hydrophilicity, nano-SiO_2 can be categorized into hydrophilic and hydrophobic types [12]. In the context of concrete applications, hydrophilic nano-SiO_2 is predominantly used due to its excellent dispersion capabilities in water. This dispersion characteristic is the primary reason for its preferred usage in concrete [13].

The advent of nanomaterials has brought about exciting opportunities and advancements in membrane processes. As concerns grew over the rapid depletion of clean water resources, researchers began exploring numerous modifications in membrane-nanomaterial composites, expanding their potential for various application. Therefore, this paper is aimed to give the critical review about the usage of SiO_2 in membrane technology.

2 Synthesis Methods of SiO_2-based Membrane

There are several methods used to synthesize Polymer-SiO_2 based membrane. **Table 1** summarize synthesis methods and SiO_2 used in membrane.

Table 1. Synthesis methods for Polymer-SiO_2 based membrane

Methods	Matrix	NPs	Ref
Bulk-modification	Polyphenylene oxide (PPO) - SiO_2	Hydrophobic nanosilica (10-25 nm)	[14]
Coating	Mullite-zeolite-alumina-SiO_2	nanosilica (25-35 nm)	[15]
Electrospinning	Polyvinylidene fluoride-co-trifluoroethylene chloride (PVDF-CTFE)/ SiO_2	Nanosilica 40nm	[16]
Electrospinning-Electrospraying	Polyvinylidene fluoride (PVDF)/SiO_2	Hydrophobic nanosilica (40 nm)	[17]
Electrospinning	Polyimide (PI)/SiO_2	Hydrophobic nanosilica (10-50 nm)	[18]
Phase Inversion	Polysulfone (PSF)/ Polyvinylpyrrolidone (PVP)/SiO_2	Nanosilica (10-20 nm)	[19]
Coating	Copper ferrocyanide (CuFC)/SiO_2/PVDF	Nanosilca (300 nm)	[20]
Phase inversion	Polyethersulfone (PES)/graphene oxide (GO)-SiO_2	Nanosilica (20 nm)	[21]
Nonsolvent-induced Phase Inversion	PES-(SiO_2-g-polymethacrylic acid (PMAA))	Nanosilica (10-80 nm)	[22]
Phase Inversion	Polyacrylonitrile (PAN)-SiO_2	Nanosilica (23 nm)	[23]

PPO-SiO_2 membrane which contained 2 wt% of SiO_2 is synthesized through bulk modification [14]. **Figure 1** shows SEM image of membrane. membrane shows inner finger-like pores and a sponge porous sub-layer. It is evident that the presence of silica reduces the number of finger-like pores in membrane This discrepancy can be attributed to the impact of SiO_2 particles during the preparation step, affecting solvent/non-solvent exchange. From the surface we can see that the addition of SiO_2 decreasing the pore size of membrane.

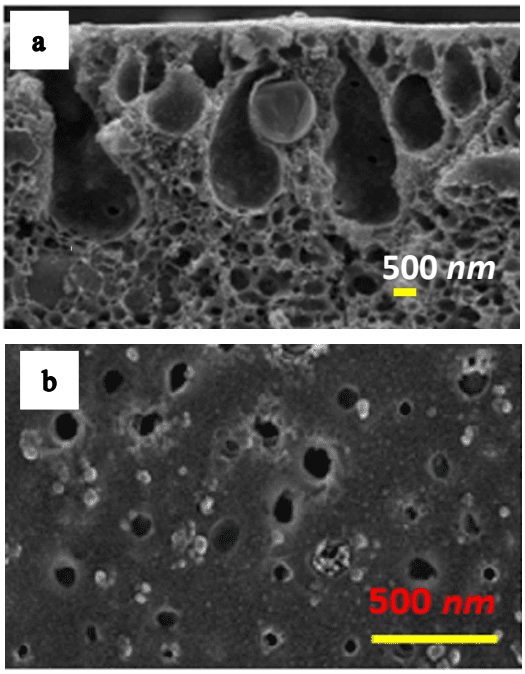

Figure 1. SEM Image of PPO-SiO_2 membrane (a) cross-section (b)surface [14].

Meanwhile, Jafari et.al [15] synthesized mullite-alumina-zeolite (MAZ) membrane coated with 2 g of hydrophilic SiO_2. Morphology of membrane shown at **Figure 2**. a flat, smooth, and crack-free surface was observed on the membrane after the formation of the SiO_2 layer

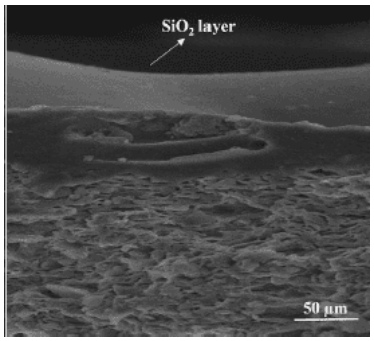

Figure 2. SEM Image of MAZ-SiO_2 membrane (surface) [15].

PVDF-CTFE/SiO_2 NFs membranes were synthesized by Ren et.al in a single electrospinning step using the optimal spinning parameters determined in previous experiments. SiO_2 nanoparticles were first dispersed in the DMF: acetone (7: 3, m: m) solvent using an ultrasonic treatment for 30 min to ensure uniform dispersion. Then, a constant quantity of PVDF-CTFE was added to the pre-treated solvent mixture. After 12h of vigorous stirring at 60˚C and 30 min of ultrasonic treatment to eliminate bubbles, a homogeneous precursor solution was produced. Lastly, electrospinning was performed using the same spinning parameters as those used in the preceding experiments. Morphology of membranes are shown in **Figure 3**. From the image, it was evident that the fiber surface became rough due to the addition of SiO_2 nanoparticles. A micro-convex structure resembling a lotus leaf surface was formed as the number of protrusions on the fiber surface increased with the added amount. In addition, the addition of SiO_2 nanoparticles increased the spinning solution viscosity and the average diameter of the resulting fibers. Clearly, the instability of the spinning fluid caused by the viscosity changes gradually widened the fiber diameter distribution.

Another method introduced by Yan et.al. [17], they were synthesized membrane through electrospinning-electrospraying method. Electro-spinning solution was made of PVDF 14 wt% in DMAc and the electrospraying solution was made of 5 wt% of PVDF and 2 wt% of SiO_2. **Figure 4** depicted the surface morphology of the prepared micro/nanospheres. It can be clearly observed that with the increase of SiO_2 content, in the deposited PVDF/SiO_2 micro/nanospheres, the PVDF polymer changed from a dense structure to a loose porous structure, and more and more SiO_2 particles attached to the surrounding of PVDF structure. The SiO_2 particles formed a protrusion structure on the surface of micro/nanospheres. The results showed that SiO_2 particles as physical barrier can effectively reduce the contact between PVDF molecular chains, so as to form the micro/nanospheres with porous PVDF structure as the skeleton and SiO_2 as the filler.

Meanwhile, Alkindy et. al [21] synthesized PES/GO-SiO_2 membrane through phase inversion method. PES/GO-SiO_2 membranes were each prepared by using a loading concentration of 1.0 wt% of the respective nanoparticle to the polymer. The corresponding nanoparticles were dispersed in DMAc and ultrasonicated in a water bath for 30 min. PVP (4%) was dissolved in the above solutions followed by the addition of PES (16%) and stirred for 24 hours at 60 ºC. The dope solution was set aside for 24 hours to remove trapped air bubbles (i.e. membrane degassing). The solution was subsequently cast on a polyester membrane support on clean glass at a thickness of 200 µm.

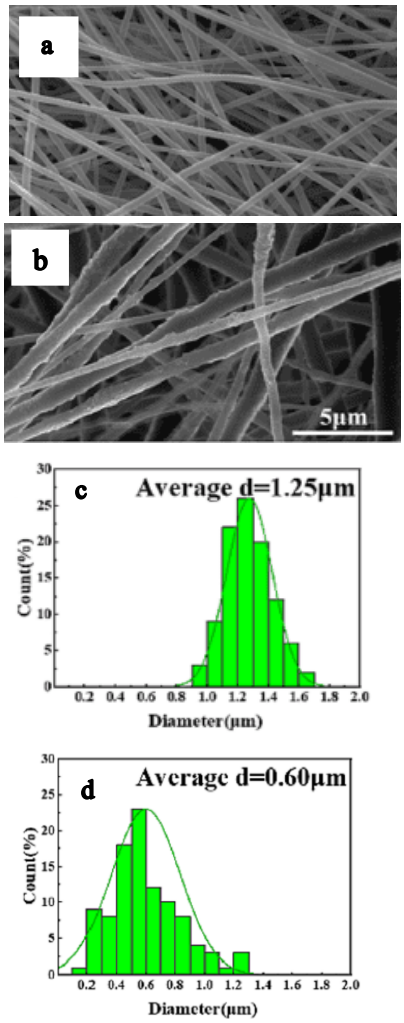

Figure 3. SEM image and particle distribution of (a, c) PVDF/CTFE and (b, d) PVDF/CTFE/SiO_2 membranes [24].

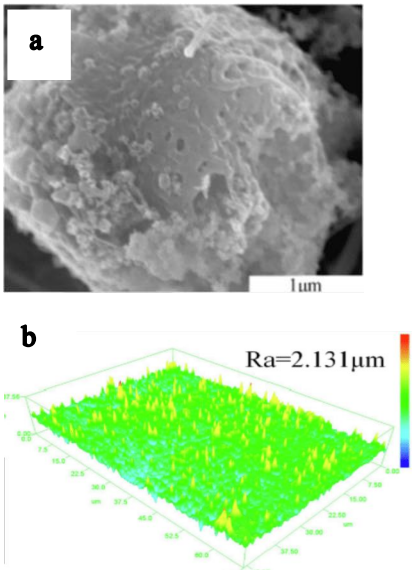

Figure 4. (a) SEM image and (b) surface roughness of PVDF-SiO_2 membrane [17].

The glass plate was immersed horizontally into deionized water at a temperature of 25 ºC for 24 hours. Finally, the membranes were washed with DI and stored for use. A control PES membrane was also prepared using the same method for comparison. SEM image of membrane, porosity and hydrophilicity of membrane are shown in **Figure 5**. the formation of macrovoids was clearly visible. This could be explained by the synergistic effect of the GO/SiO_2 leading to increased hydrophilicity of the nanocomposite. This increase in hydrophilicity increases the exchange rate between the solvent and nonsolvent in the coagulation process, resulting in the formation of macrovoids or increased porosity. the addition of SiO_2 and/or GO nanoparticles in the PES matrices decreased the contact angle and improved hydrophilicity. The contact angle decreased from 85º in the pristine PES membrane to 58º in the PES/GO-SiO_2 MMM. Contact angle measure-ments indicate the hydrophilic/hydrophobic nature of the membrane. For oil-in-water emulsions, hydrophilic/ oleophobic membranes are preferred as they allow water to pass through while rejecting oil.

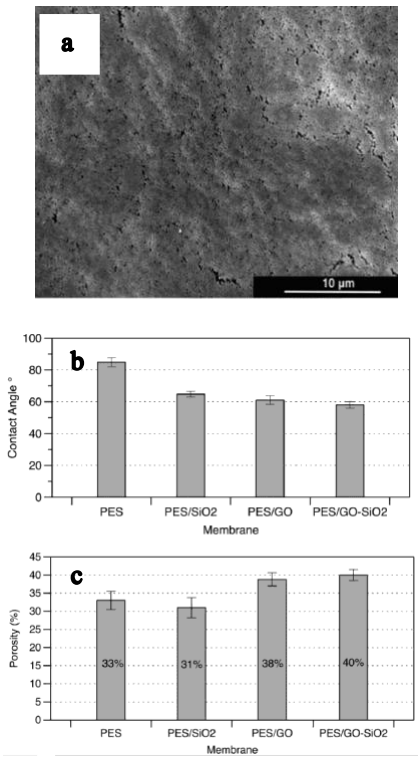

Figure 5. (a) *S*urface SEM image, (b) contact angle and (c) porosity of PES/GO-SiO_2 [21].

3 Recent Applications of SiO_2-based Membrane

Table 2 shown the recent application of SiO_2 based membrane.

Table 2. Recent application and performance of SiO_2 based membranes

Membranes	Applications	Perfor-mance	Ref
Polyphenylene oxide (PPO) -SiO_2	CO_2 separation	CO_2 absorpstion flux 5.8 × 10-3 mol. $m^{-2}s^{-1}$ and removal efficiency up to 64.8 %	[14]
Mullite-zeolite-alumina-SiO_2	Oil-water Emulsion separation	Water flux ~690 ($kg/m^2.h$) COD rejection up to 99.2%	[15]
PVDF-CTFE/SiO_2	Oil-water Separation	Separation efficiency up to 99%	[16]
PVDF/SiO_2	Oil-water emulsion separation	Separation efficiency up to 99.4% Permeability 110 $L^{-1}m^{-2}h^{-1}$	[17]
PSF/PVP/ SiO_2	Amoxicillin removal from aqueous solution	Flux and rejection of amoxicillin up to 90%	[19]
CuFC/SiO_2/ PVDF	Selective removal of Cesium	Flux 669 $L^{-1}m^{-2}h^{-1}bar^{-1}$ and rejection up to 99.99%	[20]
PAN-SiO_2	Ultrafiltration	Water flux 384 ± 20 LMH and rejection of BSA 98.6 ± 2%	[23]

The performance of PVDF-SiO_2 was observed. It was observed that the water-in-oil emulsion fed before separation was opaque and milky, while the filtered liquid obtained after separation was transparent and clear. When magnifying the opaque and milky liquid, many small droplets could be seen, namely water-in-oil droplets. While, almost no droplets were observed in the magnified view of filtrate, indicating that membrane could effectively separate the water-in-oil emulsion. Then the reusability was detected by 10 cycles of water-in-oil (kerosene) emulsions separation experiment. After each cycle test, the membrane was washed by ethyl alcohol for 3.0 min and dried for next use. After 10 cycles, the separation efficiency of membrane remained above 99.40%, and the flux was stable above 110.0 $L^{-1}m^{-2}h^{-1}$, indicating that membrane had good reusability in the separation process of water-in-oil emulsion. After each cycle, the flux of the membrane could be recovered by simple cleaning, indicating that the membrane had good resistance for fouling. Other works used SiO_2 for oil separation due to their multi-layer and porous characteristics, mostly had a separation efficiency of >98% [25].

Hydrophobic silica can be used in After coating of SiO_2 and CuFC, the flux of modified membrane decreased by 88% and 86% compared with pristine membrane at the SiO_2 concentration of 0.1% and 0.5%, respectively. The hydrophilicity and flux of the membrane increased with increasing the concentration of SiO_2. Therefore, the membrane with 0.5% SiO_2 exhibited higher water flux than that with 0.1% SiO_2. Although the flux of 0.1% SiO_2 loading CuFC/SiO_2/PVDF membrane was lower than that of the 0.5% SiO_2 loading membrane, XPS and SEM results indicated that oversaturated membrane with 0.5% SiO_2 might lead to instability of CuFC loading layer. Therefore, the membrane structure was more stable at the suitable amount of SiO_2 for supporting. The residual concentration of Cs was lower than 0.2 μg L^{-1} during 6 h filtration, which could reduce radioactivity from high (> 0.8μg L^{-1}) to intermediate level. Therefore, to maximize the membrane efficiency, the 0.1% SiO_2 loading CuFC/ SiO_2/PVDF membranes was applied for the next experiments. The membrane filtered water after 1 h and 8 h was sampled, and the silicon content was analysed by ICP-MS after it was digested by 5% HF. The results shown that the silicon concentration was lower than the detection limit of ICP-MS, indicating that the leaching amount of SiO_2 was negligible and the purified water could not get contaminated with toxic SiO_2.

The results of amoxicillin separation by the synthesized nanocomposite membranes containing of SiO_2 nanoparticles and the pure

Polysulfone membrane are shown in **Figure 6**. Accordingly, the percentage of amoxicillin removals for M-0 to M-4 membranes were 66.52, 71.23, 79.82, 82.86 and 89.81%, respectively. As it is evident, all PSF/PVP/SiO_2 membranes exhibit more rejection efficiency compared to pure PSF membranes. In addition, the amoxicillin rejection for nanocomposite membranes increased with increasing SiO_2 nanoparticles. Accordingly, the M-4 membrane could reduce the amoxicillin concentration from 30 mg/L to 3 mg/L in the permeate. Thus, with about 90% separation efficiency, the highest rejection efficiency was obtained. But M-4 membrane revealed less pure water flux than M-3. The mechanism for the removal of organic molecules (here, amoxicillin) by modified membranes can include some mechanisms such as size exclusion (sieving, steric effect), electrostatic discharges (electrical, Donnan) and adsorption on the membrane surface. The separation efficiency may be increases due to blocking of the pores or narrowing the pores by precipitating the pollutant molecules. The size exclusion mechanism can partially prevent the passage of amoxicillin molecules through membrane pores. Also, blocking some of the pores of the membrane by amoxicillin molecule (steric effect) can increase the efficiency of separation.

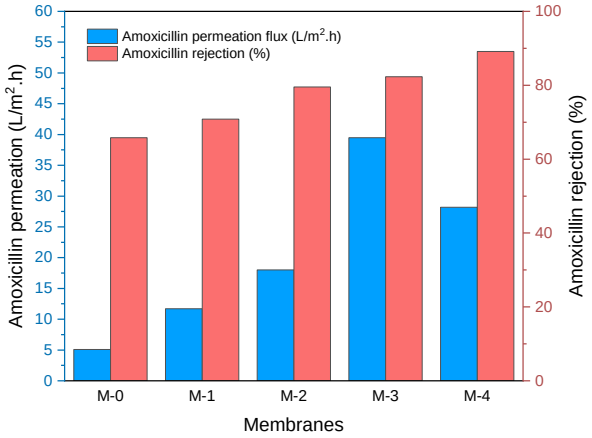

Figure 6. performance of PSF/PVP/SiO_2 membrane [19].

Another hydrophobic silica used in CO_2 separation. The performances are shown in figure 7. it is evident that the CO_2 absorption flux significantly increases from 9.5×10^{-4} ($molm^{-2}s^{-1}$) for neat sample to 5.5×10^{-3} ($molm^{-2}s^{-1}$) for M-5Et. Increase of porosity is the main reason for CO_2 absorption flux increment, in the other words: the higher the porosity, the higher the flux. In fact, the contact area between gas and liquid phase is enhanced by high porosity. Decreased in membrane resistance against gas transport is favourable for the CO_2 absorption. The porous membranes in membrane contactor process are not selective and just provide high interfacial zone for contact between gas and liquid phases. Thus, the removal efficiency substantially depends on the interaction between liquid absorbent and gas components. Other works shown that the permeability of the membrane to H_2 was 100 barrer and the H_2/CO_2 separation coefficient reached 13.30 [26].

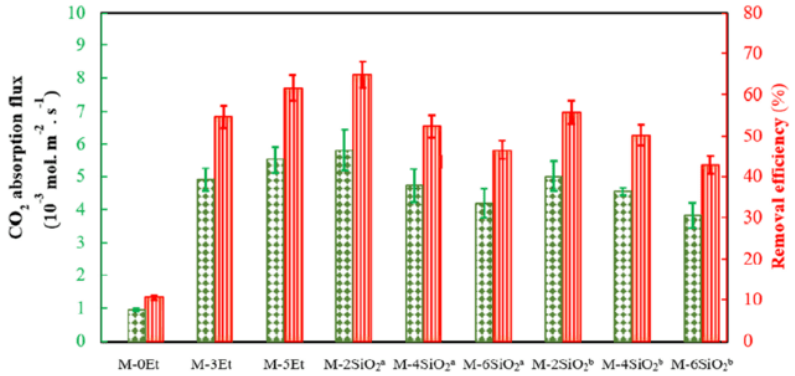

Figure 7. the performance of CO_2 absorption for various PPO/SiO_2 membrane (24).

The performance of PVDF-CTFE/SiO_2 were observed. It was observed that the membrane with SiO_2 2wt% has the highest permeability flux and separation efficiency of higher than 99%. The NFs membrane could not only effectively separate the CCl_4-water mixture and emulsion but also diesel, n-hexane, and petroleum ether as the oil phase mixture and emulsion. The analysis of the filtrate's water content revealed that the membrane had an exceptional separation effect on various types of oil-water mixtures and emulsions. After separation, the water content of the emulsion and mixture was less than 150 ppm and 50 ppm, respectively. In addition, CCl_4, which has a higher density than water, exhibited the highest permeate flux, whereas diesel, which has a lower density than water and a greater viscosity, exhibited the lowest permeate flux. This demonstrated that when oil and water were separated under gravity, the oil permeate flux was proportional to the oil density and inversely correlated with the oil viscosity. Furthermore, the oil-water separation performance of the obtained membrane was compared with those of NFs membranes made of other materials. It was found that the separation flux and efficiency of the PVDF-CTFE/SiO_2 NFs membrane obtained in this work were both higher than other membranes under gravity conditions. Other works shown that the addition of SiO_2 could enhance NF membranes for salts rejection up to 98.45% [27].

4 Future Challenge of SiO_2 in Membrane Technology

Nano concrete shows excellent performance because nano-SiO_2 has a large surface area. At the same time, some problems in the research have also received the attention of scholars. For example, although high specific surface area makes concrete show more excellent performance, nano-SiO_2 aggregates because of its high specific surface area, which affects the dispersion effect in water [28]. However, the widespread use of nano-adsorbents such as nano-silica is often restricted by the easily occurred nanoparticle agglomeration leading to low efficiency [29]. For instance, increasing the pH, nanoparticle concentration, and calcium content in a solution containing silica nanoparticles has been reported to lead to higher silica aggregation [30]. Therefore, physical and chemical dispersion techniques have been developed to improve the dispersion of nanomaterials.

Physical strategies for providing improved nanoparticle dispersion include long term mechanical stirring, ultrasonication, and the use of physical dispersants. In comparison to these physical methods, chemical dispersion techniques usually lead to superior nanoparticle dispersion [31]. This is because chemical dispersion methods are less affected by the applied shear force detaching physically adsorbed dispersants and high ionic concentrations suppressing the electrostatic repulsion force during operation [32]. The introduction of ultrasonic technology improves the dispersion of nano-SiO_2 in water, but it is still an important direction of improving the dispersion effect of nano-SiO_2. In addition, the colloidal silica sol containing monodisperse nanoparticles is easy to form floccules and coatings on the surface of cement particles after combining with cement. These floccules can retain more free water, which is more obvious than the improvement of the hydration effect of nano-SiO_2 [33]. Therefore, the application of colloidal silica sol in concrete deserves attention.

5 Conclusion

This review is discussing about the synthesis, recent application and the future challenge of SiO_2 in membrane technology. It can be concluded that mostly SiO_2 used in membrane has the size between 10-300 nm with various corporation method to the membrane, which is bulk modification, coating, electrospinning, and phase inversion. The recent application of SiO_2 membrane are various. for oil-water separation, the addition of SiO_2 could reject oil up to 99.4%, for gas separation, the addition of SiO_2 could absorb CO_2 up to 64.8% and other application that shows the better performance after the addition of SiO_2. The dispersion of SiO_2 in the matrix could be the future challenge of SiO_2-based membrane.

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