The Fabrication of Oleophobic Coating and Its Application in Particulates Filtration

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Abstract: The stir-frying process in Chinese cooking has produced serious emissions of oily particles, which are an important source of urban air pollution. In particular, the complex composition of fine particulate may pose a threat to human respiratory and immune systems. However, current filtration methods for oily particulate fumes have low filtration efficiency, high resistance, and high equipment costs. In polypropylene (PP) electret filters, efficiency rapidly decreases and pressure drop (wind resistance) sharply increases after the adsorption of oily particles, due to the oleophilic properties of the PP fibre. We addressed this issue of filter performance degradation by fabricating a sodium perfluorooctanoate (SPFO) oleophobic coating on polyvinylidene fluoride (PVDF) fibre membranes for oily particle filtration. The SPFO coating showed a promising oleophobic effect even at low concentrations, which suggests it has oleophobic properties for different oil types and can be modified for different substrates. This fabricated oleophobic coating is thermostable and the oleophobic effect is unaffected by temperatures from 0 to 100 °C. By modifying the SPFO coating on the PVDF membrane, a high filtration efficiency (89.43%) and low wind resistance (69 Pa) was achieved without oil adhesion, so the proposed coating can be applied in the filtration and purification of oily fine particles and offers a potential strategy for preventing atmospheric oil pollution.

Keywords: oleophobic coating; sodium perfluorooctanoate; PVDF fibre; oily particles filtration

1. Introduction

Stir-frying in Chinese cooking produces a large amount of oily particulate matter during the cooking process, and the cooking fume emissions have been an important source of urban air pollution in recent years [1,2]. The composition of cooking fumes is very complex, and can cause significant environmental pollution and health hazards. Numerous studies have shown that cooking fumes are genotoxic [3], reproductive-toxic [4], carcinogenic [5–7], and mutagenic [8], and can present a serious threat to the human respiratory and immune systems. The fine particulate fumes containing oily fine particles can lead to asthma [9], bronchitis [10], emphysema [11], pulmonary fibrosis [12], and even lung cancer [13,14]. In addition, fine particulate fumes can cause blockage of dermal pores, cardiovascular diseases, and germ-cell carcinogenesis [15]. Therefore, the filtration and prevention of oily particles, especially oily fine particles, is of great significance.

Many experts from various fields have conducted research on the treatment of oily fumes. Current approaches to filtering oily particles include mechanical separation, activated carbon adsorption, wetting treatment, electrostatic treatment, and fabric filtration. Among these, mechanical separation is widely used in industrial processes, requiring simple equipment and causing a small drop in airflow pressure. However, the removal
efficiency of oily fumes, especially of fine and ultrafine particles, is low, and its equipment requires frequent maintenance and has high energy consumption [16]. The activated carbon adsorption method has higher removal efficiency, but the filters have to be replaced frequently because the pressure drop sharply increases [17]. The wetting treatment method can be used to remove fumes using water spray, which can be subject to serious wastewater contamination [18]. Electrostatic treatment has high filtering efficiency due to electric coagulation effects, by which fume particles are collected on a plate by electric adhesion. However, the very high cost of equipment maintenance limits its application [19].

The fabric filtration method has been preferred in recent years due to its easy usage and excellent ability to filter oily fine particles. This method captures fume particles by using a fabric filter, and has obvious advantages, such as having simple equipment, mild conditions, easy operation, and high filtration efficiency, and being low-cost [18]. However, the fabric filters, especially those made of the commonly-used polypropylene (PP) material, are mostly oleophilic, which means their pressure drop increases and efficiency decreases after absorbing oil [20,21]. Hence, these filters require regular replacement, resulting in both high energy consumption and high costs, which limit their wide application. New techniques and materials must be considered to minimise this pressure drop without influencing the filtration efficiency.

Oleophobic materials are widely adopted for oil-water separation [22–24]. For example, hydrophilic and underwater oleophobic coatings or structures were applied on PVDF membranes. Polydopamine [25] and polyphenolic-amine [26] coatings and hollow structures [27,28] were studied and used to obtain underwater oleophobic properties. However, there have been few studies focusing on oleophobic filters used for air filtration. An oleophobic coating may provide a new way to resolve the problem by using the low surface energy of the chemical used for coating. The fine and ultrafine oily particles from fumes are captured by the filter fibres, and the small captured oily droplets bond with one another, forming bigger oil droplets. The bigger oil droplets then fall from the fibres due to the oleophobic effect of the coating [29], without affecting filtration efficiency or pressure drop.

In this study, a sodium perfluorooctanoate (SPFO) coating was used to form a polymer/fluorinated surfactant complex with substrate materials. In our previous works, we have developed filtering fabrics [30–35], of which polyvinylidene fluoride (PVDF) has proven to be a substrate material with high efficiency, low resistance, and reduced energy consumption [35]. Filters with modified PVDF have advantageous piezoelectric and pyroelectric effects, resulting in a nanoscale filter membrane that can trap fine particles via electrostatic effects, in addition to traditional mechanisms such as diffusion, inertial collision, and interception [36]. This electrostatic force can capture not only charged particles, but also neutral polarised particles, by electrostatic polarisation. PVDF nanofibre fabrics were adopted as the substrate instead of traditional fabric filters.

Currently, oleophobic coatings are usually made with polyelectrolyte/fluorinated surfactant, fluorosilanes, fluorinated copolymers, and hydrogels. Among these, fluorosilane is commonly used on multi-porous surfaces, where it adheres to and fills the pores [37], hence sharply increasing the pressure drop. Fluorinated copolymers are complex in composition and fabrication, preventing them from being applied in industrial production [38]. Polyelectrolyte/fluorinated surfactant complexes form through synergistic zipper-like interactions between polyelectrolyte chains and oppositely charged fluorinated surfactant molecules [39]. This arrangement places the hydrophilic portion of the fluorinated surfactant in the near-polyelectrolyte surface region by electrostatic attraction, while the fluorinated alkyl chains are oriented toward the air-solid interface to provide low surface energy, typically in the range of 6–18 mN/m [40].

Accordingly, the SPFO coating has low total polarisation and low polarity [41], thus exhibiting low intermolecular attraction, and therefore having oleophobic properties. Furthermore, the coating is chemically inert and thermally stable [42] due to the high electronegativity of the carbon-fluorine bond. Therefore, the adoption of the PVDF fibre membrane
as the substrate, modified with a SPFO oleophobic coating, will allow for filtration of oily fine particles that is efficient, convenient, and has low resistance.

2. Materials and Methods

2.1. Materials

Polyvinyldiene fluoride (PVDF, average molecular weight 640,000) was supplied by the Solvay Corporation, Alpharetta, GA 30005, USA. Perfluorooctanoic acid (PFOA) was supplied by Shanghai Aladdin Bio-Chem Technology Co., Ltd. DFM (Beijing Chemical Industry Group Co., Ltd., 2 Huaibaishu St., Beijing, China) and EA (Beijing Tong Guang Fine Chemicals Company, Beijing, China) were used as received. The blended, soybean, sesame, and sunflower oils were all commercially available.

2.2. Preparation of Oleophobic Coating Materials

Specific amounts of PFOA and sodium hydroxide solids were weighed according to the required mass fractions (0.001%, 0.005%, 0.01%, 0.05%, 0.1%, 0.15%, 0.2%, and 0.25%), then 10 mL of ethanol was added to the solid mixture. The mixture was stirred until completely dissolved and fully reacted to produce the different mass fractions of the SPFO ethanol solution.

2.3. Preparation of SPFO Coated PVDF Fibre Membranes

The PVDF membrane was prepared as described in our previous work [35]. The SPFO solution was sprayed onto the PVDF membrane using a spray gun, and dried at room temperature (25 °C) to allow ethanol to evaporate completely; this was followed by rinsing with a small amount of deionised water. The SPFO ethanol solution was tested at different mass fractions (0.001%, 0.005%, 0.01%, 0.05%, 0.1%, 0.15%, 0.2%, and 0.25%), and the 0.005 wt.% SPFO ethanolic solution was chosen for our experiment.

2.4. Static Contact Angle Measurement

To test the oleophobic performance of the SPFO coating, the static oil contact angle was measured by an automatic contact angle measuring instrument (DSA30, KRÜSS) at a temperature of 25 ± 1 °C, relative humidity of 50% ± 5%, and oil droplet size of 6 ± 1 µL. The surface oleophobic filter had a contact angle of over 120° with all the different types of oil. Therefore, the tests that were not specifically mentioned were performed with blended oil.

Static oil contact angles under different temperatures were tested by placing the SPFO-coated filter on a heating pad. Once the filter was stabilised at a certain temperature, the oil contact angles were tested using the method described above, allowing the oleophobic effect of the coating to be tested at different temperatures.

2.5. SEM Characterisation

The surface morphologies of filters before and after the SPFO coating were characterised by scanning electron microscopy (SEM, SU8000, Hitachi, Tokyo, Japan). SEM characterisation was carried out on each of the aforementioned filters to ensure complete coverage of the oleophobic SPFO coating.

2.6. Filtration Performance Characterisation

The filtration performances of the filters before and after the SPFO modification were characterised by a standard process, during which standard salt particulates (0.3 µm NaCl) and standard oily fine particulates (0.3 µm DEHS) are filtered using a particulate matter detector (TSI 8130) at an airflow of 32 L/min. By comparing the filtration performance of the unmodified PVDF membrane with that of the SPFO-coated PVDF membrane, the effect of the SPFO oleophobic coating could be observed. The oils adhesion simulation, which simulated the difference in filtration performance before and after the oil adhesion, showed
the enhancement in filtration performance produced by the SPFO coating in multiple oil filtration scenarios.

3. Results and Discussion
3.1. Surface Morphology of SPFO Oleophobic Coating

The SPFO oleophobic coating was layered onto PVDF fibre membranes to fabricate oleophobic PVDF fibre membranes, as shown in Figure 1a, which can be used for filtering oily fine particles. The morphologies of the PVDF fibre membrane before and after the SPFO modification, and during oil adhesion, were characterised and analysed (Figure 1b–d).

![Figure 1.](image)

Figure 1. The fabrication process and morphologies of the SPFO oleophobic coating: (a) schematic diagram of fabrication of SPFO-coated PVDF; (b,c) SEM image of uncoated and SPFO-coated PVDF fibre membranes; inserted images are of oil contact angles; (d) SEM image of oil-adhered SPFO-coated PVDF fibre membrane.

From the images shown in Figure 1b,c, the larger fibre diameter indicated that the PVDF fibre membrane was successfully modified with a complete and uniform coating. The successful modification of the sodium perfluorooctanoate coating was also evidenced by an increase in the oil contact angle, which changed from being completely lipophilic (CA = 0°) without the coating, to oleophobic (CA = 128.7°) with the SPFO coating. Moreover, there was no obvious oily particle adhesion on the SPFO-coated PVDF fibre membrane, which indicated that the SPFO coating can be used in oily particle filtration to prevent adhesion, as well as to intercept and filter oily fine particles by applying it to smaller pore size filters.

3.2. Oleophobic Effect of SPFO Oleophobic Coating

Many factors, such as the concentration of SPFO, types of oil droplets and substrates, temperature, and duration at high temperatures, can influence the oleophobic effect of the SPFO coating. The effect of the concentration of SPFO with different mass fractions of SPFO ethanolic solutions was characterised by oil contact angle tests, as shown in Figure 2a. The oil contact angles of PVDF fibre membranes modified with different mass fractions of the SPFO ethanolic solution (0.001%, 0.005%, 0.01%, 0.05%, 0.1%, 0.15%, 0.2%, and 0.25%) were compared with those of the unmodified PVDF fibre membranes. The results showed that consistent oleophobic effects could be obtained, with oil contact angles of about 120.0° when the mass fractions reached 0.005 wt.% and above. Therefore, a SPFO ethanolic solution with
0.005 wt.% was chosen for the following experiments, considering practical applications and cost.

![Graph](image)

**Figure 2.** The oleophobic effect of the SPFO coating under different conditions. The oil contact angles of SPFO-coated PVDF fibre membranes with (a) different SPFO concentrations and (b) different oils. The oil contact angles of SPFO-coated stainless steel mesh at (c) different temperatures of 30 and 100 °C and (d) with increasing exposure time at 100 °C.

We investigated the filtration effects of different types of oil that are typically involved in cooking fume emissions [43], and the influence of oil droplet types on the oleophobic effect are shown in Figure 2b. The results showed that the SPFO coating was effective in preventing adhesion for the commonly used oils. Furthermore, different substrates, including PVDF fibre membranes, polypropylene (PP) fibre membranes, industrially used filters, stainless steel mesh, and brass mesh, were modified in the same way with the selected 0.005 wt.% SPFO ethanolic solution. These substrates were modified in the same way, and the oil contact angles were all above 120.0°.

As oily fumes are produced under high-temperature conditions, it is important to investigate how temperature affects the oleophobic performance of SPFO-coated filters. The SPFO coating was also modified on stainless steel mesh, which is insensitive to temperature changes, to monitor the oil contact angles at different temperatures, as shown in Figure 2c. The results indicated that the impact of temperature on the SPFO-coated stainless steel mesh was negligible in the measured temperature range, i.e., 0–100 °C. In addition, there was no significant change in the oil contact angles with increasing exposure time at 100 °C (Figure 2d), meaning that the surface oleophobic effect was not affected by prolonged exposure to oil droplets, even at a high temperature.

However, when the substrate was PVDF fibre membranes, temperature had an obvious impact on the contact angle. The oil contact angles of the SPFO-coated PVDF fibre
membrane were monitored under different temperatures (as shown in Figure 3a). The temperature did not have an obvious impact on the oil contact angles ranging from 0 to 45 °C, but when the temperature increased from 45 to 100 °C, the oil contact angles sharply decreased. However, the oil contact angles returned to their original values when the temperature was lowered again. This indicates that the influence of temperature on the SPFO-coated PVDF membrane is reversible. In addition, the oil contact angles became progressively smaller as the exposure time increased at 100 °C (Figure 3b), which indicates that the surface of the PVDF fibre membrane changes from an oleophobic state to a super-oleophilic state after prolonged exposure to high temperatures.

Figure 3. The oleophobic effect and its reversibility of SPFO-coated PVDF fibre membranes under different temperatures. (a) The oil contact angles of SPFO-coated PVDF fibre membranes at different temperatures. (b) The oleophobic effect at exposure time at 100 °C. (c) The reversible oleophobic-lipophilic switching mechanism of SPFO-coated PVDF fibre membranes with temperature change.

To further understand the surface-oil interaction of the SPFO-coated PVDF membranes above 45 °C, we propose the theoretical model in Figure 3c to explain the switching mechanism from oleophobic to lipophilic properties. This phenomenon can be explained by the fact that the perfluorooctanoic anionic chain is in mobile status and is reorganised on the substrate surface [29]. This reorganisation allows molecules passing through the temporary defects of the fluorinated outermost layer to penetrate the lipophilic PVDF membrane, causing the surface-oil interaction switching mechanism.
3.3. Filtration Performance of SPFO-Coated PVDF Fibre Membranes

The filtration efficiency and airflow resistance (pressure drop) of the SPFO-coated PVDF fibre membranes are needed to estimate the filtration performance of oily fine particulates. The filtration efficiency and airflow resistance were measured at a 32 L/min airflow with reference to the standard filter testing process, and the results are shown in Table 1.

Table 1. The filtration efficiency and airflow resistance of PVDF filters at 32 L/min airflow.

| Filter                      | Salt Fine Particulate Matter | Oily Fine Particulate Matter |
|-----------------------------|------------------------------|------------------------------|
|                             | Filtration Efficiency (%)    | Filtration Efficiency (%)    |
|                             | Airflow Resistance (Pa)       | Airflow Resistance (Pa)       |
|-----------------------------|------------------------------|------------------------------|
| Original PVDF               | 99.41                        | 98.37                        |
| 26                          | 26                           |                              |
| Oil-accumulated PVDF        | 30.66                        | 24.74                        |
| 220                         | 225                          |                              |
| SPFO coated PVDF            | 94.12                        | 89.43                        |
| 69                          | 69                           |                              |
| Oil-accumulated SPFO coated | 74.99                        | 76.06                        |
| PVDF                        | 69                           | 82                           |

The original PVDF fibres had excellent filtration efficiency and airflow resistance. However, when oily particles accumulated on the PVDF fibres, the filtration efficiency sharply decreased from 99.41% to 30.66%, while the airflow resistance increased from 26 to 220 Pa. This drastically limits its application in filtering oily particles. The SPFO-coated PVDF fibre membrane achieved filtration efficiencies of 94.12% and 89.43% for salt (0.3 μm NaCl) and oily fine particulate matter (0.3 μm DEHS), respectively, with an airflow resistance (pressure drop) of 69 Pa. Following oily particle accumulation on the SPFO-coated PVDF fibres, the filtration efficiency slightly dropped; however, the airflow resistance stayed the same. These results indicated that compared with uncoated PVDF, the SPFO-coated PVDF fibre membrane can achieve filtration of oily fine particles with higher efficiency and lower resistance, even after oil accumulation, making it a promising solution for industrial applications.

To characterise the stability and reusability of the SPFO coatings, cleaning treatments were conducted using commercially available cleaning agents. After several cleaning treatments, the oleophobicity of the coating exhibited a slight decrease, as shown in Figure 4. The excellent wear-resistance and easy cleaning properties make SPFO coatings suitable for oily particle filtration.

Figure 4. The properties of SPFO coating after several cleaning treatments. (a) The morphologies and oil contact angles of SPFO coatings after 10 to 40 cleaning treatments using cleaning agents. In SEM images, SPFO coating was prepared on stainless steel mesh to show wear on coating surface; (b) the oil contact angles of SPFO coating at different cleaning times.
4. Conclusions

To summarise, oleophobic coatings were successfully fabricated for different substrates, and they exhibited excellent adhesive properties with the substrates. The coated fibres showed obvious oleophobicity for several kinds of oils, with oil contact angles higher than 120°. The SPFO-coated PVDF fibre membranes had high filtration efficiency and relatively low airflow resistance for 0.3 µm oily fine particulates, and were almost unaffected by continuous oil accumulation. The stable oleophobicity under higher temperatures and ease of reusability make SPFO coatings feasible for application under practical fume emission conditions. To conclude, the ease of fabrication, high filtering performance, stable oleophobicity under higher temperatures, and long life span of oleophobic coatings make them feasible for use in the filtration of fume emissions, for individual face masks, and hair protection.

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