Fast fabricating cross-linked nanofibers into flameproof metal foam by air-drawn electrospinning for electrostatically assisted particle removal

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ABSTRACT
Air filtration is of vital importance for creating healthy breathing indoor air. Particle filters can cause relatively high air resistance and large energy consumption in building ventilation systems. Electrostatically assisted metal foam (EAMF) filters have been developed to provide relatively high efficiency with low air resistance. This study reported a method of fast fabricating cross-linked PVDF nanofibers inside nickel foams by air-drawn electrospinning. The improved EAMF-PVDF filter is flameproof, which provides high security in practices. The new filter increases the absolute efficiency for PM 0.3-0.5 (particulate matter between 0.3 and 0.5 μm in aerodynamic diameter) of the bare EAMF filter by 15% and keeps an average efficiency of 83.1% for 28-day filtering outdoor air at the filtering velocity of 1 m/s. Besides, the daily efficiency fluctuation of EAMF filter shrank from 27.3% to 3.4% after adding nanofibers. The fabricated nanofibers can be cleaned off by burning when reaching the life span, and then a repeated fast fabrication can regenerate the high efficiency. This work provides a new route to combining electrostatic stable polymer nanofibers with flameproof metal foam for fabricating air filters with high efficiency, low air resistance and fire resistance.

1. Introduction
Particulate matter (PM) is harmful to human health [1,2]. At present, PM pollution is still severe in developing countries like China and India [3–6], and sometimes wildfire smoke caused by forest fires worldwide also contains enormous PM [7]. What’s worse, it has been proved that PM pollution has close contact with influenza and respiratory diseases [5,8,9]. Since the coronavirus disease (COVID-19) pandemic hit the world earlier in 2020, this disease outbreak has spread rapidly over almost all the countries and areas, resulting in over 110.7 million confirmed cases, including 2.46 million deaths globally by February 21st, 2021 and is still ongoing [10]. The aerosol transmission has been recognized as a possible pathway of SARS-CoV-2 [11–13]. From the air samples collected in Wuhan Fangcang hospitals, Liu et al. [14] identified that SARS-CoV-2 RNA was mainly carried by particles of 0.25–0.5 μm and 0.5–1 μm. Therefore, to keep humans safe from PM pollution and virus infection, it is vital to control airborne particle concentration in building ventilation systems for healthy indoor environments.

High-efficiency particulate air (HEPA) filters are widely used to prevent people from PM pollution efficiently. HEPA filters can cause large energy consumption due to the relatively high air resistance if used in building ventilation systems [15]. The energy consumption of ventilation fans driving filters accounts for up to 50% of building ventilation systems [16]. Electrostatic precipitator (ESP) is another commonly used method to remove airborne PM [17,18]. The air resistance of ESP is much lower than that of HEPA filters. However, the dust holding capacity is limited by the area of dust collection plates, and the ozone production by electrical discharge may bring health risks [19,20]. In a word, it is a challenge to achieve high-efficiency filtration with low air resistance and large dust holding capacity by only one kind of filtration technology.

Several studies have been carried out to overcome the challenge. Many researchers fabricated nanofibers to raise the filtration efficiencies for fine and ultrafine particles. Electrospinning, a viable technique for generating ultrathin fibers, was used to produce such nanofibers [21]. Liu et al. developed polyacrylonitrile (PAN) nanofibers, which had a one-pass efficiency of 95% for PM 0.3 (particulate matter under 2.5 μm in aerodynamic diameter) at the air face velocity of 0.21 m/s [22]. The material had high light transmission, but the air resistance was still large, ranging from 133 Pa to 206 Pa [22]. Wang et al. developed an...
electrospun nanofiber made of polyvinylidene fluoride (PVDF) / polytetrafluoroethylene (PTFE), which had a one-pass efficiency of 99.97% for PM$_{2.5}$ [23]. Meanwhile, the air resistance was 57 Pa at the air face velocity of 0.053 m/s [23]. Wang et al. developed a highly efficient transparent air filter by a bipolar electrospinning apparatus [24]. It can reduce the air resistance from ~295 Pa to ~240 Pa compared with the single electrode electrospinning when the nanofiber filters reached the efficiency of 99.1% for PM$_{2.5}$ at the air face velocities of 0.5-0.6 m/s [24]. If used in building ventilation systems at a high air velocity of 2-3 m/s, the air resistances of above-mentioned nanofiber filters will be too large and consume large energy consumption. Therefore, some researchers applied continuous electrostatic effects to traditional filters to achieve high filtration efficiency with lower resistance. [25–27]. Feng et al. developed a pin-filter medium-grounded conductive plate structure to continuously charge the filter, which observably enhanced the efficiency from 29% to 97% for particles of 0.4 μm under 25 kV charging [27,28]. The resistance was 38 Pa at the air face velocity of 0.1 m/s [27,28]. Tian and Mo developed an electrostatically assisted air (EAA) filter, which can increase the efficiency for PM$_{0.3,0.5}$ of a polyethylene terephthalate (PET) coarse filter from 0.4% to 99% by particle pre-charging and filter polarizing [26]. The resistance was 21 Pa at 1.2 m/s air face velocity. A follow-up study showed that additional loading of dielectric materials on PET filters would significantly generate abundant charges on filter surfaces, which would lead to a strong attraction to precharged particles and result in high filtration efficiency [29].

Nevertheless, PET filters and many other polymer filtration materials are vulnerable to fire [30,31]. The electrostatic discharge produces possible sparks, which may cause a fire in buildings. An electrostatically assisted filtration technology based on metal foam has been proposed to solve this problem since metal foams are usually well flameproof [32]. The electrostatically assisted metal foam (EAMF) coarse filter can reach 78.9% efficiency for 0.3–0.5 μm particles with 10.8 Pa resistance at 0.5 m/s air face velocity. However, in the previous studies, the long-term performance of EAMF filters has not been reported. Therefore, the long-term performance and the life span of EAMF filters need to be further tested and discussed.

This study combined polyvinylidene fluoride (PVDF) nanofibers with the EAMF filter by electrospinning to improve filtration performances. We measured the filtration efficiency, air resistance, net ozone increase and flammability of the advanced EAMF-PVDF filter, and compared them with those of the bare EAMF filter. We investigated how the voltages and metal foam pore size affect filtration performances. A long-term test was carried out to identify the life span of this new flameproof EAMF-PVDF filter.

2. Methodology

2.1. Fabricating nanofibers in metal foams

2.1.1. Air-drawn electrospinning setup

Fig. 1 is the schematic of the air-drawn electrospinning setup. The apparatus consists of a handheld electrospinning device (MPEG-1, JUNADA, China), a hollow steel plate as a conductive collector and a centrifugal fan (130FLJ1, JF-MOTOR, China). A syringe pump (LSP01-1A, LONGER, China), which provides the spinning solution, is connected to the electrospinning device. The centrifugal fan (130FLJ1, LONGER, China), which provides the spinning solution, is connected to the positive and grounded electrodes of an internal high-voltage direct-current (HVDC) power supply, respectively. The centrifugal fan is set to draw the air through a metal foam on the top of the plate. The spinning solution synchronously driven by the electric force and air drag force goes into the metal foam, forming cross-linked nanofiber networks.

2.1.2. Materials

Spinning solution preparation

The spinning PVDF solution was prepared according to Kang’s study [33]. PVDF powder (FR915, density 1.76 g/cm$^3$, purity 99.5%) was provided by Shanghai 3F New Materials Co., Ltd., China. The dissolvent was a mixture of acetone and N,N-Dimethylformamide (DMF) with a volume ratio of 2:3. Acetone (purity 99.5%) was supplied by Beijing Chemical Works, China, and DMF (purity 99.5%) was provided by Shanghai Titan Scientific Co., Ltd., China. PVDF powder was mixed with the dissolvent at a mass ratio of 1:4. The mixed solution was stirred in a magnetic stirrer at 500 r/min for 15 min at room temperature (24–26 ℃).

Metal foams

Table 1 shows the properties of nickel metal foams used in this study. The nickel foams with the size of 80 mm × 80 mm × 1 mm were provided by Tengerhu Electronic, China. The porosities of metal foams were described in ppi (pores per inch), which designated the pores' number in one linear inch. The purity of metal foams were all over 99%.

2.1.3. Electrospinning process

In the electrospinning process, the target nickel foam was fixed on the top of the hollow steel plate. The internal electrospinning power supply voltage was set as 10 kV, and the injection speed of the syringe pump was set as 7 ml/h. The air velocity was set at 1 m/s through the nickel foam and the plate. The handheld electrospinning device was moved slowly and uniformly to make nanofibers depositing into the nickel foam. During the process, the distance between the spinneret and the plate was 100 mm. The electrospinning process was 2 min for each nickel foam.

2.2. Performance test

Fig. 2 is the schematic setup to test the electrostatically assisted filtration performance of the prepared nickel foams. The test duct was a small-scale acrylic air duct with a cross-sectional area of 80 mm × 80 mm. A frequency conversion centrifugal fan was used to introduce
outdoor air with particles into the air duct. The airflow velocity was controlled at 1 m/s. The main filtration section included the charging part and the collecting part [29]. In the charging part, the tungsten charging pin matrix was connected to a 0 to −30 kV adjustable HVDC power supply (N30, GENVOLT, China), in front of which was a hollow steel plate attached to the ground. The hole diameter in the hollow steel plate was 20 mm. The vertical distance between the charging pin and the hollow steel plate was 7.5 mm. The schematic and physical charging part were shown in Fig. A1 in Appendix A. The collecting part consisted of two hollow steel plates with a distance of 5 mm. The upstream plate was connected to another 0 ~ +30 kV adjustable HVDC power supply (P30, GENVOLT, China) while the downstream plate was attached to the ground. The nickel foam with nanofibers was fixed in the middle of two plates in parallel. A sensor was used to monitor temperature and
humidity in the air duct. The number concentrations of PM$_{0.3-10}$ (particulate matter between 0.3 and 10 μm in aerodynamic diameter) were measured by a particle counter (Aerotrak 9306, TSI Inc., USA) and the ozone concentration was recorded by a photometric ozone monitor (Model 205, 2B Tech., USA) at the upstream and downstream of the filtration section. The mass concentration data of outdoor PM$_{2.5}$ were obtained from a website database (www.pm25.in/beijing). A differential gauge (DP-CALC 5825, TSI Inc., USA) was used to measure the pressure drop across the filtration section.

The one-pass efficiency for particles in specific diameter was calculated as:

$$\eta = \left(1 - \frac{C_{up}}{C_{down}}\right) \times 100\%$$

where, $\eta$ is one-pass efficiency; $C_{up}$ and $C_{down}$ are the number concentrations of particles at the upstream and downstream of the filtration module, respectively.

The net ozone increase in the whole device was calculated as:

$$\Delta C_o = C_{o,down} - C_{o,up}$$

where, $\Delta C_o$ is the net ozone increase of the module; $C_{o, up}$ and $C_{o, down}$ are concentrations of ozone (ppb) at the upstream and downstream of the filtration module, respectively.

Power dissipation $P$ (W) was calculated as:

$$P = U_1 \cdot I_1 + U_2 \cdot I_2$$

where, subscripts 1 and 2 stand for the charging zone and collecting zone, respectively; $U$ is the power supply voltage (kV); $I$ is the loop current (mA). Both $U$ and $I$ were recorded by the HVDC power supply.

A referential parameter called efficiency growth coefficient ($\varepsilon$, W$^{-1}$) was defined to determine the optimal charging voltage before turning on the collecting part.

$$\varepsilon_x = \frac{\eta_{x+1} - \eta_x}{P_{x+1} - P_x}$$

where, $\eta$ is the efficiency; $P$ is the power cost (W), W. The subscripts $x$ and $x + 1$ present the charging voltage. This coefficient indicates how much efficiency growth per unit power cost can be achieved at a specific charging voltage.

3. Results and discussion

3.1. Characteristics of nickel foam with PVDF nanofibers

The photos of nickel foams before and after electrospinning are shown in Fig. A2. Besides, we observed nickel foams with nanofibers by an optical microscope (SK2208, SAIKE Digital, China). As shown in Fig. 3 (a) and (b), the space between nickel fibers with nanofibers was filled by some transparent fibers, scattering light under 100× magnification. The electrospun PVDF fibers were well-knitted together with the nickel foam deeply when observed under 400× magnification, while the black shadow around was the nickel foam structure in Fig. 3 (c). The nanofibers can increase much more deposition area for the bare nickel foam. Thus, the EAMF-PVDF filter is supposed to provide a longer life span than the bare EAMF filter.

We also observed nickel foams with nanofibers by a scanning electron microscope (SEM; Merlin, Zeiss, Germany). As shown in Fig. 4, PVDF nanofibers can be seen to form nets between the large nickel foam holes. Fig. A3 displays the just prepared electrospun nanofibers with PVDF knots. The electrospun nanofibers can capture plenty of particles, as shown in Fig. 4 (b) and (c).

3.2. Filtration performance of EAMF-PVDF filter

The physical and electrical characteristics of EAMF-PVDF filters

![Fig. 4. SEM images of 75 ppi nickel foams before and after electrospinning. (a) Bare nickel foam (b) Nickel foam spun with nanofibers (after particle filtration, 1000 X) (c) Nickel foam spun with nanofibers (after particle filtration, 2000 X).](image)
would change after electrospinning, which may influence the electrostatically assisted filtration efficiency. Fig. 5 shows the filtration efficiency of EAMF-PVDF filters for PM$_{0.3-0.5}$ (particulate matter between 0.3 and 0.5 μm in aerodynamic diameter) and net ozone increase. The currents of power supplies were determined by charging voltages. The nickel foam used in this experiment was Foam #1 (40 ppi), and the air face velocity was set at 1 m/s. The loading PM were outdoor ambient particles. The air temperature and moisture content were measured as 20.4–27.3 °C and 1.74–3.68 g/kg (dry air). The range for number concentration and the size distribution of inlet particles are shown in Fig. A4.

As supposed, charging voltage enhanced filtration efficiency. Since the pore size of 40 ppi metal foam was as large as 0.425 mm, the filtration efficiency was nearly 0% when there was no charging electric field, as shown in Fig. 5. With charging voltage increasing from 0 to ~10 kV, the efficiency grew up to 64.6%. Meanwhile, at the expense of pre-charging electrostatic enhancement, the power supply current and ozone concentration change increased to 0.132 mA and 55.8 ppb, respectively. However, the efficiency growth coefficient, $\varepsilon$, was only 0.065 W$^{-1}$ at ~9 kV, as shown in Fig. 5. It indicates less benefit in filtration efficiency when increasing the charging voltage higher than ~9 kV. Therefore, we set the charging voltage at ~9 kV in the following experiments.

As shown in Fig. 6, the one-pass efficiency increased to 78.9% after the collecting voltages from 0 to 15 kV. When the collecting voltage increased from 0 to 5 kV, the net ozone increase of the whole device decreased from 52.3 ppb to 44.7 ppb on the contrary. The reason might be that more ozone was dissociated on the nickel foam or consumed by oxidizing the nickel foam as enhancing the collecting voltage [32,34,35]. However, as the collecting voltage further increased from 5 to 15 kV, the net ozone increase hardly grew. The increasing current produced more ozone indeed, while the enhanced electric field also made the nickel foam dissociate or consume more ozone at the same time. The results reveal a balance between the increase and decrease of ozone caused by collecting voltages.

Fig. 7 shows that the absolute values of electrostatically-assisted efficiency increases of Foams #1 ~ #3 after electrospinning are 11.9%, 16.9% and 23.0%, respectively. When the porosity increased, the efficiency growth of electrospinning nickel foams increased. However, the air resistance and energy consumption would also increase with the porosity increasing. Therefore, considering the balance between filtration efficiency and energy saving, 75 ppi nickel foam was chosen as the primary material in the following studies and applications.

To evaluate the effect of nanofiber loading on the long-term performance of EAMF filters, the metal foam performances before and after electrospinning were both continuously tested for five days. As discussed in Section 3.2, 75 ppi nickel foam was selected for the control experiments. The charging voltage was set as ~9 kV and the collecting voltage was +18 kV. During the test, the device was operated 24 h a day. Across the entire trial, the concentration of outdoor PM$_{2.5}$ in the EAMF-PVDF filter group was slightly higher than that in the EAMF filter group. Besides, in the two groups, the average temperature and
humidity were close, and the charging and collecting voltages were the same. The results are shown in Fig. 9.

The one-pass efficiency of the bare EAMF filter for PM$_{0.3-0.5}$ decreased rapidly from 75% to 43% in 5 days. The daily efficiency fluctuated within a wide range. To take the third day as an example, the upper quartile of efficiencies was 75.1% while the lower quartile was 47.8%; thus, the device’s stability was flawed. On the contrary, as for the EAMF-PVDF filter, the initial efficiency was 15% higher than the bare EAMF filter, and the efficiency was maintained at 87.1% (the average value of 453 points measured in five days) within five days. There was no efficiency decrease as shown in Fig. 9. The upper quartile of efficiencies in five days was 89.4% while the lower quartile was 85.1%, so the efficiency fluctuation range in each day was small, which means the stability of the EAMF-PVDF filter was better than that of the bare EAMF filter in the long run. Under the air face velocity of 1 m/s, the air resistance of the EAMF filter was 4 Pa while the EAMF-PVDF filter was 21 Pa. However, 21 Pa was still much lower than that of HEPA filters. Besides, the net ozone increase of the device changed from 43 ppb to 79 ppb after electrospinning. The reason might be the nanofibers coated around the metal foam structure hindered the ozone consumption of nickel.

After the 5-day performance test, we continued to operate the EAMF-PVDF filter 24 h a day with the same experimental parameters to evaluate the stability of the high efficiency.

It can be seen in Fig. 10 that the EAMF-PVDF filter kept a high efficiency of 83.1% (the average value of 2598 points measured in 28 days) in 4 weeks. Besides, the upper quartile of efficiencies in 28 days was 84.8% while the lower quartile was 81.4%, so the efficiency fluctuation in each day was small. We stopped the test on the 28th day because the device’s air resistance increased to as large as 228 Pa. Therefore, we recommend that the nickel foam with nanofibers in this filter module be cleaned after continuous 28-day use.

The flammability of traditional polymer foam filters and EAMF-PVDF filters were tested. We lighted up a commonly-used polyurethane filter and an EAMF-PVDF filter, as shown in the video in Supplementary material B. The video and Fig. 11 indicated that the polyurethane filter was easily lighted up and completely burned down in seconds, while there was no flame when burning the EAMF-PVDF filter. In practice, the PVDF nanofibers in the EAMF-PVDF filter can be cleaned off by burning when reaching the life span, and then a repeated fast fabrication can regenerate the high efficiency of the EAMF-PVDF filter. Fig. A5 in Supplementary material A shows the continuous test results of the EAMF-PVDF filter after a repeated fast electrospinning.
fabrication. During the test, the efficiency was 82.4% on average with a little fluctuation in about 80 h, which matches the results in Fig. 9 and Fig. 10 very well. It indicates that the EAMF-PVDF filter can be renewed by burning and repeated fast fabrication.

4. Conclusions

This study used air-drawn electrospinning to fabricate cross-linked PVDF nanofibers inside nickel foams fast. The new EAMF-PVDF filter overcame two critical disadvantages of the bare EAMF filters: the efficiency decreases rapidly and fluctuates significantly in the long run. The deeply loaded nanofibers inside nickel metal foam increased the absolute efficiency for PM$_{0.3-0.5}$ of the bare EAMF filters by 15%, and kept a high efficiency of 83.1% for 28-day filtering outdoor air at the filtering velocity of 1 m/s, while the efficiency of the bare EAMF filter efficiency decreased from 75% to 43% in 5 days. The daily efficiency fluctuation of the EAMF filter shrank from 27.3% to 3.4% after electrospinning. The EAMF-PVDF filter is well flameproof and is more suitable for high-security-required indoor environments [26,37]. The PVDF nanofibers in the EAMF-PVDF filter can be cleaned off, and then a repeated fast fabrication can regenerate the high efficiency of the EAMF-PVDF filter. This work provides a new route to combining electrostatic stable polymer nanofibers with flameproof metal foam for fabricating air filters with high efficiency, low air resistance and fire resistance. The new filters can be used in broad areas, including buildings, subways and industries.

CRediT authorship contribution statement

Fanxuan Xia: Methodology, Formal analysis, Resources, Data Curation, Writing - Original Draft, Writing - Review & Editing. Yilun Gao: Methodology, Resources. Enze Tian: Methodology, Writing - review & editing. Alireza Afshari: Visualization, Writing - review & editing. Jinhan Mo: Conceptualization, Methodology, Project administration, Funding acquisition, Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary material A: Charging part of the EAMF-PVDF filter (Fig. A1). Comparison between nickel foam before and after quick fabrication of PVDF nanofibers by electrospinning (Fig. A2). SEM image of 75 ppi nickel foam after electrospinning (Fig. A3). The range for number concentration and the size distribution of inlet particles in the performance test of the EAMF-PVDF filter (Fig. A4). One-pass filtration efficiencies of PM$_{0.3-0.5}$ for the renewed EAMF-PVDF filters (Fig. A5).

Supplementary material B: Video of Burning Comparison between polyurethane filter and nickel foam fabricated with PVDF nanofibers. Supplementary data to this article can be found online at https://doi.org/10.1016/j.seppur.2021.119076.

References


