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# Preparation of Electrospun Composite PEG/PVP Phase-Change Nanofibers and their Application in Cigarette Cooling Filters

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**Abstract**
Composite phase-change nanofibers were prepared by electrospinning with polyethylene glycol (PEG) as the phase-change material (PCM) and polyvinylpyrrolidone (PVP) as the carrier matrix. The high PEG content endowed the nanofibers with an excellent cooling effect and significantly reduced the smoke temperature. For PEG70/PVP nanofibers, the smoke temperature can be decreased 45℃ at the 8th puff, and be kept below 45℃. The cooling test proved that the electrospun PEG/PVP phase-change nanofibers exhibited a desirable cooling performance, improving the comfort of cigarette products. And the composite PEG/PVP phase-change nanofibers present great potential as the cooling cigarette filter material for HnB tobacco application.

**Keywords**
Electrospun nanofiber, Phase-change material, Cigarette cooling filter, PEG/PVP PCM.

## 1. Introduction

When a traditional cigarette is ignited, the temperature can reach as high as 600-900 °C, and many harmful substances are released by the burning pyrolysis, which brings harmful and/or carcinogenic effects to the health of smokers and the environments. Therefore, there has a rapid development in tobacco products in recent years, and new tobacco products have been constantly produced. Among them, heat-not-burn tobacco (HnB) products attract a lot of research interest because their smoking characteristics are closest to those of traditional cigarette products. For HnB products, the atomization medium and aroma components in the tobacco substance are heated to 250 °C to 350 °C to release atomized smoke. The distillation and pyrolysis products of the shredded tobacco are produced in a non-combustible state, and the harmful products released can be greatly reduced. However, the smoke temperature of HnB is significantly higher than that of the traditional cigarette when the smoke enters the mouth due to the large heating area and the single way of heat dissipation, and smokers will feel a burning sensation and an unpleasant aftertaste, which greatly affects the smoking experience of consumers.

In some cigarette products the length of the filter rod is increased to decrease the smoke temperature, but the increase in the length of the filter rod will increase the user's suction resistance during the suction process and affect costumers' relaxed feeling when smoking. Moreover, a hollow filter rod or a complex structure design are made to make the smoke travel a longer gas path in the filter rod to decrease the smoke temperature of the HnB cigarette. However, the cooling effect is very limited due to the limited length and diameter of the cigarette filter rod.

The other popular technology is to cool the mainstream smoke by adding heat-absorbing materials. For example, folded polylactic acid (PLA) film is used as the cooling material for the smoke in iQOS (I-Quit-Ordinary-Smoking) supporting cigarettes, launched by Philip Morris, but the PLA film is easy to collapse and shrink when heated, affecting the cooling effect, and the sensory evaluation of the taste [1]. Ammonium salt is used as a cigarette cooling agent, which can significantly reduce the temperature of the smoke; but a small amount of ammonia gas will be generated when the ammonium salt is heated, which brings a pungent odor and health risks [2]. Moreover, endothermic gel is also added into the cooling filter stick to decrease the smoke temperature, but this gel can melt into a liquid state in the flue gas temperature range, causing the risk of leaking from the filter stick and affecting consumers' vaping experience [3]. Therefore, it is still a challenge to develop a cigarette cooling material that can effectively reduce the smoke temperature and does not affect the smoking experience.

Because the cigarette application scenario is more complex, it is difficult for a single cooling method to achieve the ideal stable effect. However, multiple components with different functions can be added to the composite material which can not only improve the thermal conductivity of the composite material, but also solve the leakage problem during the phase transformation of the heat-absorbing material. This paper aims at solving the above-mentioned problems in the HnB cigarette. We prepared functional composite phase-change fibers, in which polyvinylpyrrolidone (PVP) was used as a polymer carrier to avoid the collapse of the filter rod; and polyethylene glycol (PEG) as a phase change polymer to reduce the smoke temperature. During the suction process, the PEG/PVP nanofibers can effectively reduce the flue gas temperature at the mouth end of the filter rod, and can solve the overflow problem that may occur due to melting of the phase change material, thereby improving the suction experience.

## 2. Materials and Methods

### 2.1. Materials

Polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP, K30, average molecular weight - 40000); chloroform; dimethylacrylamide were purchased from Sinopharm chemical reagent Co. Ltd. (Shanghai, China). All reagents were used directly without further purification.

### 2.2. Methods

Firstly, 5 g of PVP was dissolved in ultra-pure water to form a PVP solution with a mass fraction of 50%, then PEG with different relative molecular weights and contents was added and stirred for 12 hours.

The PEG/PVP solutions were loaded into a syringe and the injection needle replaced with a metal needle. The speed of the automatic syringe pump was set to 1ml/h, and an aluminum foil was placed at a distance of 15 cm to the metal needle to collect the PEG/PVP electrospun fibers. The ambient temperature was 27 °C and the relative humidity was 55%. The PEG/PVP solution was injected into the metal needle, and the nanofibers formed were collected on the aluminum foil under a voltage of 18 kV. All the PEG/PVP electrospun fibers were collected and dried in a vacuum oven at 60 °C for 24 hours to remove the residual organic solvent.

## 3. Results and Discussion

### 3.1. Preparation of PEG/PVP nanofibers of different diameter

PCMs are functional materials that can release or absorb heat through reversible phase transitions to maintain the temperature of the system [4, 5]. They have excellent heat storage and temperature adjustment properties and can be used in clothing, construction, military, and many other fields [6-9]. However, PCMs are unstable and prone to leakage during the phase transition process, which severely limits their application in the field of energy storage. Electrospinning technology [10, 11] is a feasible method to encapsulate PCMs in support materials to prepare PCM@ polymer fibers [12, 13].

PEG is a kind of phase change material frequently used in HnB cigarettes. Low, medium and high molecular weight PEG are mixed and sprayed on the filter tow in a certain proportion; PEG can also be mixed with hexanol. The mixture can be made into a granular state, round strip shape or coating state heat-absorbing gel and added to the filter rod, which can reduce the flue gas temperature by 1-8 °C [14].

PEG/PVP electrospun nanofibers were collected under a voltage of 18kV, and the receiving distance was 15 cm. PEG with different relative molecular mass was used to prepare the phase-change nanofibers. When the relative molecular weights of PEG were 8000, 10000 and 20000, the corresponding diameters of the composite fibers obtained were 100 nm, 180 nm and 600 nm, respectively (Fig.1). As the relative molecular weight of PEG in the mixed solution increased, the viscosity of the solution and the average diameter of the composite fiber increased correspondingly.

To test the cooling effect of the phase-change nanofibers, PEG/PVA nanofibers were added in a sandwich rod with a thickness of about 2 cm (Fig. 2). A fast miniature temperature-measuring thermocouple was introduced at a distance of 2 mm from the front and back of the interlayer and at the center of the cooling material, and the temperature measurement point of the thermocouple was located at the center of the filter tip. The temperature changes of manually lighted cigarette smoke were recorded under standard conditions at the temperature measurement points throughout the process. The purpose of short-distance temperature measurement was to reduce the influence of the environment and individual differences in filters on temperature measurement.

### 3.2. Effect of fiber diameter on cooling effect

From Fig. 1, we can see that the average diameter of electrospun composite nanofibers clearly increased with an increase in the relative molecular mass of PEG, and the cooling effect of 3 kinds of PEG/PVP phase-change nanofibers on the flue gas temperature were tested. The results are shown in Fig. 3, When the fiber diameter increased from 100 nm to 180 nm, the temperature decreased slightly by 3 °C, with 180 nm nanofibers showing a better cooling effect. But when the fiber diameter increased from 180 nm to 600 nm, only a 0.5 °C degree difference was observed, indicating that the diameter of nanofibers showed little effect on the cooling performance of the phase-change nanofiber filter.

### 3.3. Cooling effect of phase-change nanofibers of different mass fraction

We investigated the cooling effect of the PCM content of phase-change nanofiber on the smoke temperature at the outlet of heated cigarettes, the results of which are shown in Fig. 4. It can be seen that when the PEG content increased from 30% to 50%, PEG/PVP=50:50, the maximum smoke temperature at the outlet of the heated cigarette dropped from 60 °C to 45 °C, and the temperature drop was as high as 15 °C, indicating that the amount of PCM used can significantly reduce the temperature of the flue gas. However, when the PEG content increased from 50% to 70%, the maximum outlet flue gas temperature only decreases by 2 °C. These results indicated that PCM endows PEG/PVP nanofibers with heat storage and temperature regulation capabilities. The larger the amount of PCM fed, the greater the latent heat value of PEG/PVP fibers, and the more obvious the heat storage and temperature regulation effect.

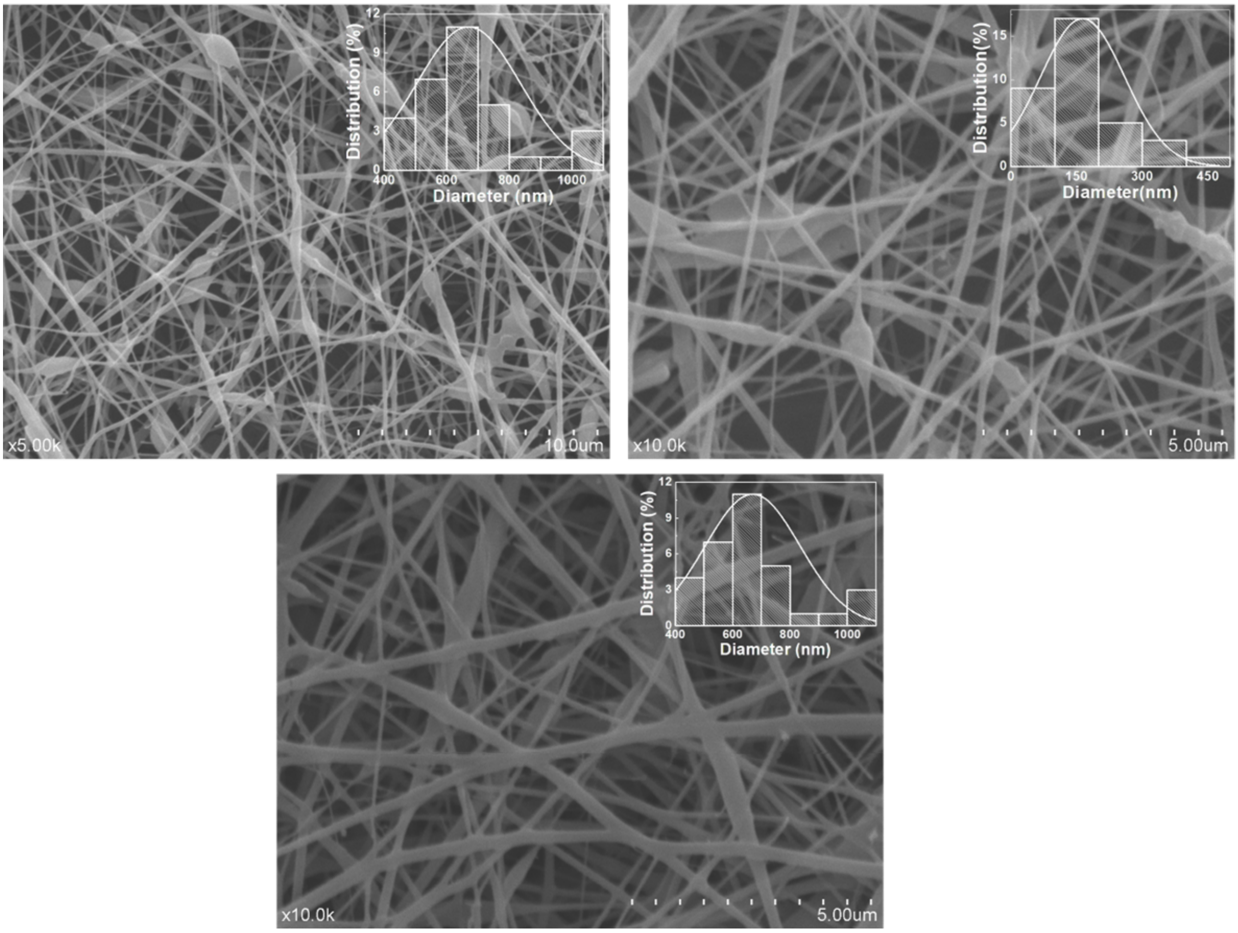


*Fig. 1. SEM images and the diameter distributions of PEG/PVP electrospinning fibers of different PEG molecular weight. A) PEG 8000, B) PEG 10000, C) PEG 20000*

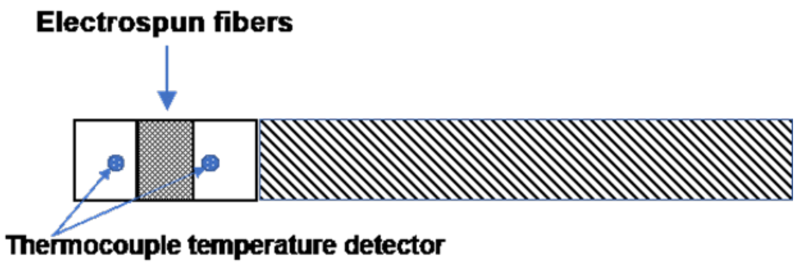


*Fig. 2. Schematic illustration for temperature measurement method for phase-change nanofibers cigarette filter temperature*

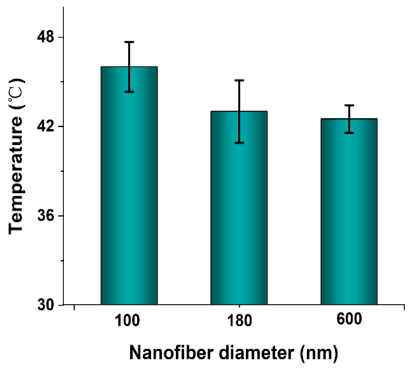


*Fig. 3. Cooling effect of composite phase-change nanofiber cigarette filter of different diameter*

## 3.4. Effect of the length of phase-change nanofibers

In order to investigate the effect of the length of the cooling section on the cooling performance of the filter rod, the same PEG/PVP phase-change nanofiber section and cigarette section were used, and only the cooling section and the empty tube section (total length of 26 mm) of an ordinary cigarette were replaced with 30, 25 and 20 mm nanofibers, respectively. The results are shown in Fig. 5. It can be seen that the longer the cooling section is, the lower the outlet flue gas temperature will be. The outlet smoke temperature was 15 and 12 °C lower than that of the ordinary cigarette, respectively. Even with the cigarette with the shortest cooling section (20 mm), the outlet smoke temperature was 8 °C lower than that of the ordinary cigarette. The contact area of the flue gas increased for the longer nanofiber filter to improve the cooling effect.

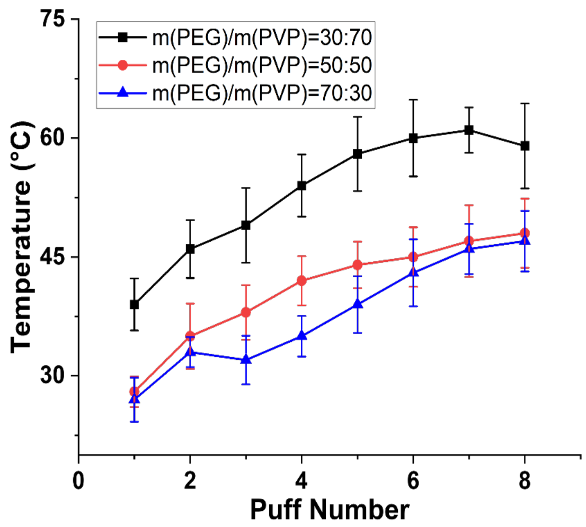


*Fig. 4. Cooling effect of the composite phase-change nanofibers of different mass fraction*

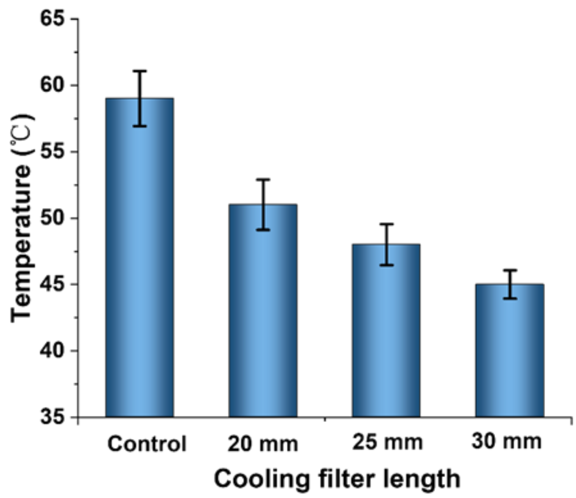


*Fig. 5. Cooling effect of composite phase-change nanofiber filter of different filter length*

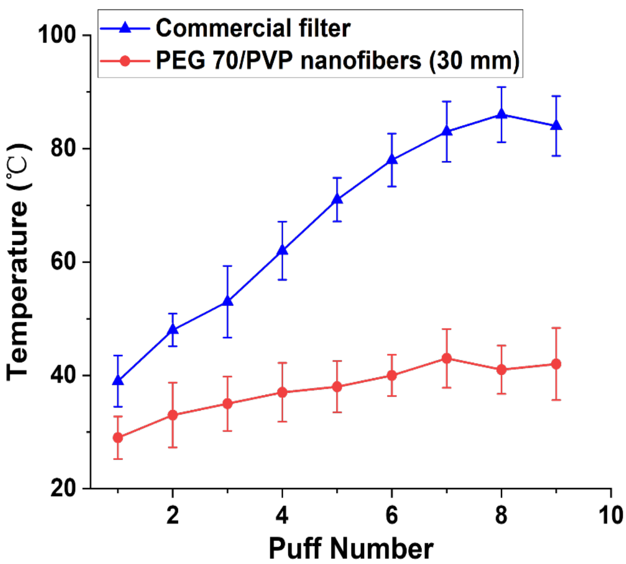


*Fig. 6. Temperature change at the rear end of the filter*

### 3.5. Cooling effect of composite phase-change nanofiber filter stick compared with a commercial cigarette.

During the 9 puff smoking experiment, two thermocouples on both sides of the middle layer added along with a cigarette cooling agent were used to measure the temperature. The temperature at the outlet of the filter rod is shown in Fig. 6.

It can be seen that the mainstream smoke temperature of the commercial filter increased greatly, reached a maximum of 86 °C at the 8th puff, and then decreased to 84 °C at the 9th puff. While for the filter rod containing PEG/PVP phase-change nanofibers smoked to the first 6 puffs, the smoke temperature at the outlet of the filter was below 40 °C, which then increased to 43 °C at the 7th puff, but next decreased to 41 °C at the 8th puff and to 42 °C at the 9th puff. The mainstream smoke temperature during the whole smoking process was much lower than that of the commercial cigarettes, and the highest mainstream smoke temperature did not exceed 45 °C. The mainstream smoke temperature of the control group cigarettes was as high as 84 °C, while the oral cavity, esophagus and gastric mucosa generally tolerated temperatures of 50-60 °C, which would make smokers feel burning and irritation. Whereas the composite PEG/PVP showed an excellent cooling effect, avoiding burning harm and improving the sensory quality of cigarettes.

## 4. Conclusion

In summary, the composite PEG/PVP phase-change nanofibers were prepared successfully by electrospinning, and by adjusting the PEG content, nanofibers of different diameter can be obtained. In the composite nanofibers, PEG worked as PCM effectively, decreasing the temperature of the main stream of heating smoke, along with PVP as the carrier to construct a shaped phase change material which can not only improve the thermal conductivity of PCM, but also effectively inhibit the overflow of liquid PCM. High

PEG content endows PEG/PVP fibers with temperature regulation capabilities: the larger the amount of PCM fed, the greater the latent heat value of PCM@ PVA fibers, and the more obvious the cooling effect. The mainstream smoke temperature can be decreased to 45 °C at the 8th puff, as compared to a commercial cigarette, and it was kept below 45 °C. This composite phase-change nanofibers effectively reduce the flue gas inlet temperature, and positively promote the suction sense of consumers and the benign development of the tobacco industry. The results achieved in this study can also guide the development of other filter materials.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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