Investigation of processing factors affecting flexible heating wire by coating polyester yarns with carbon nanotubes

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Abstract. A method for preparing a flexible heating wire coated with carbon nanotubes on a surface of a polyester yarn is proposed in view of the problems of poor flexibility and water washing resistance of the existing heating wire. Based on the flexible heating wire forming system, the orthogonal test method was used to study the effects of process parameters such as solution concentration and polymer content on the formation of flexible heating wire; the prepared heating wire was subjected to resistance measurement, microscopic morphology observation, heating and washing resistance tests. The results show that the optimal parameter parameters for the preparation of flexible hot wire is A³B³C²D². In this combination, the polymer content is 15 g/L and the heating wire resistance is 180.5 Ω/cm. Moreover, the photomicrograph shows that the surface has a conductive layer composed of polymer and carbon nanotubes, which has no obvious protrusions and breaks; the surface of the parallel mesh structure can obtain a heating temperature of 52.2°C at a voltage of 12 V; the resistance of heating wire does not increase with latter washing after the first washing.

1. Introduction
With the development of science and technology and the improvement of people's living standards, the traditional functions of textiles, such as warmth, protection and decoration, have been unable to cater the needs, so people have put forward higher requirements for the additional functions of textiles [1]. It should have some addition functions like perception, feedback, regulation, information transmission and so on, meanwhile, it could meet people's needs in many aspects, such as health care, entertainment or communication [2,3]. Smart heating textiles, as a kind of smart textile, play a significant role in improving warm [4]. Compared with other types of heating textiles, low-voltage electric heating textiles use electric energy as the main heating energy, which is environmentally friendly and easy obtain and control; the electric heating textiles have fast heating speed and good durability, so they are widely used in the development of smart heating textiles [5].

In low-voltage electric heating textiles, heating elements usually are metal or metal-plated electrothermal materials, non-metal electrothermal materials, and electrothermal composite materials (electric heating films, etc.) [6,7]. Donghua University [8, 9] and the University of Manchester [10] studied the heating effect of silver-plated yarn from different aspects and confirmed the feasibility of silver-plated yarn as a heating element, but the material was hard, weighty, and poorly curved. Deng [11] and others studied the textile processability of low-pressure alloy heating wire and developed a wearable low-pressure heating jacket with heating function, but its use is limited. Yan [12] and others have made carbon fiber into a heating element, which has good electrical conductivity and heating performance, but it is costly, brittle and hardly weave. Sui et al. [13] chose graphene as a heating material...
and obtained a graphene electrothermal film after high temperature annealing, it has a fast heating rate and excellent electrothermal performance, but gas permeability is poor. Therefore, the preparation of flexible heating materials with light weight, low cost, good comfort, strong stability and good electrothermal performance is an urgent problem to be solved for smart heating textiles.

The research group proposed the preparation method of polyester/carbon nanotubes (CNTs) flexible heating wire [14], designed a uniform coating system of carbon nanotubes on the surface of the yarn and produced a polyester heating wire with heating effect initially. However, the prepared heating wire at present has some problems, such as large resistance, uneven printing and dyeing and so on. In this paper, the optimal printing and dyeing process parameters are obtained through the developed flexible heating wire forming system. The heating wire was manufactured under the optimal parameters, and a series of performance tests were conducted on the heating wire, such as the resistance measurement, the microscopic morphology observation, the heating performance and the washing resistance. The idea is to produce the flexible heating wire with better performance, which lays a foundation for the research of the subsequent heating fabric.

### 2. Polyester/CNTs flexible heating wire forming system

Polyester/CNTs flexible heating wire forming system is mainly composed of vibration printing, curing molding, yarn winding and other modules (figure 1). The vibration printing module uses acoustic vibration to drive the dyeing tank containing carbon nanotube solution according to a certain frequency, thereby achieving uniform printing and dyeing on the surface of polyester yarn by controlling the frequency of the signal; the curing molding module sends the dyed polyester yarn to the incubator for curing which is controlled by the FCD-3000 series temperature; the yarn winding module controls the winding speed of the yarn according to the process requirements by controlling the stepping motor, ensures the uniformity of the heating wire forming and winds the flexible heating wire into a roll.

![Figure 1. Schematic diagram of flexible heating wire forming system.](image)

### 3. Preparation of polyester/CNTs flexible heating wire

#### 3.1. Material

The materials used in this paper are shown in table 1.
Table 1. Material.

| Material | Specification | Quality standard | Manufacturer |
|----------|---------------|------------------|--------------|
| Polyester Carboxylated multi-walled carbon nanotubes (CNTs-006-2C) | Linear density: 800D Carboxyl group content: 2.00wt% Inner diameter: 5-10nm Outer diameter: 10-20nm Length: 10-30μm | AR | Suzhou Carbon Graphene Technology Co., Ltd. |
| Polyoxyethylene lauryl ether (Brij-30) | - | EP | Shanghai Spectrum Biotechnology Co., Ltd. |
| Myristyl sulfobetaine | - | AR | Shanghai Spectrum Vibration Biotechnology Co., Ltd. |
| Nitrile latex | Solid content: 45% PH=10 | TECH | Jingjiang Tonggao Chemical Co., Ltd. |
| Waterborne polyurethane resin | - | TECH | Guangzhou Guanzhi New Material Technology Co., Ltd. |

3.2. Preparation of solution
According to the formulation of table 2, a certain amount of carbon nanotubes are firstly put into a beaker, deionized water is added, and then stirred and dehydrated. Then polyoxyethylene lauryl ether and myristyl sulfobetaine with a mass ratio of 5:4 is added, then stirred and ultrasonic vibrated for 3 hours for use.

Table 2. Formulation of CNTs solution.

| Sample number | 1 | 2 | 3 | 4 | 5 |
|---------------|---|---|---|---|---|
| CNTs /%       | 2 | 4 | 6 | 8 | 10 |
| Polyoxyethylene lauryl ether /% | 0.2 | 0.4 | 0.6 | 0.8 | 1 |
| Myristyl sulfobetaine /% | 0.25 | 0.5 | 0.75 | 1 | 1.25 |

3.3. Polyester pre-treatment process
To make the carbon nanotubes combine with the polyester surface easier. It is necessary to pretreat polyester yarn that this preprocessing can improve the surface morphology of the polyester yarn, increase the roughness of the surface and improve the coating effect [15]. The surface of the yarn is treated by alkali reduction process. The process is as follows: the polyester yarn is immersed in a NaOH solution with a mass fraction of 5% and a temperature of 100°C for 7 min, washed and dried for use.

3.4. Preparation of flexible heating wire
On the basis of the above, the waterborne polyurethane resin and nitrile latex are added to the carbon nanotube solution in a ratio of 1:1, stirred, vibrated by ultrasonic for 1 hour and poured into the dyeing tank of the vibration printing module. Then the treated polyester yarn is immersed in the carbon nanotube solution under certain process conditions. The printed polyester yarn is sent to a curing molding module for drying to obtain a flexible heating wire. The specific process conditions are shown in table 3.

Table 3. Printing and dyeing process parameters.

| Level | CNT concentration (A)/wt% | Vibration frequency (B)/Hz | Drying temperature (C)/°C | Dip dyeing time (D)/min |
|-------|--------------------------|----------------------------|---------------------------|-------------------------|
| 1     | 3                        | 40                         | 30                        | 1                       |
| 2     | 5                        | 60                         | 40                        | 2                       |
| 3     | 8                        | 80                         | 50                        | 3                       |

3.5. Performance testing and characterization
The surface resistance of the heating wire is tested with a multimeter, each sample is measured 3 times and averaged; 5 mm heating wire is taken as a test sample, and microstructure of heating wire is observed by scanning electron microscopy. (Quanta-450-FEG+X-MAX50, FEI, Oxford, UK).

4. Results and discussion

4.1. Orthogonal test

In the preparation process of flexible heating wire, many process parameters such as solution concentration, vibration frequency, dip dyeing time and drying temperature will affect the forming effect of heating wire. In this paper, the orthogonal test method is used to study the effects of multiple factors. Select four factors and three levels, L9(3^4) for orthogonal experimental design. The concentration of carbon nanotube solution, vibration frequency, drying temperature and dip dyeing time are used as test factors. The average value of the heating wire resistance of 10 cm in length is used as a test index for orthogonal test design, the test plan and test results as shown in table 4.

| Test number | CNT concentration (A)/wt% | Vibration frequency (B)/Hz | Drying temperature (C)/°C | Dip dyeing time (D)/min | Resistance (K\(\Omega\)) |
|-------------|----------------------------|----------------------------|----------------------------|-------------------------|--------------------------|
| 1           | 3(A_1)                     | 40(B_1)                    | 30(C_1)                    | 1(D_1)                  | 26.667                   |
| 2           | 3                          | 60(B_2)                    | 40(C_2)                    | 2(D_2)                  | 25.526                   |
| 3           | 3                          | 80(B_3)                    | 50(C_3)                    | 3(D_3)                  | 24.347                   |
| 4           | 5(A_2)                     | 40                         | 40                         | 3                       | 11.239                   |
| 5           | 5                          | 60                         | 50                         | 1                       | 12.815                   |
| 6           | 5                          | 80                         | 30                         | 2                       | 11.419                   |
| 7           | 8(A_3)                     | 40                         | 50                         | 3                       | 6.567                    |
| 8           | 8                          | 60                         | 30                         | 2                       | 7.950                    |
| 9           | 8                          | 80                         | 40                         | 1                       | 5.421                    |

According to the test data of table 4, the values of \(k_1\), \(k_2\) average and extreme \(R\) are calculated according to extreme difference analysis. \(k_1\), \(k_2\), and \(k_3\) are the average values of the \(K_1\), \(K_2\) and \(K_3\) values at three levels and \(R\) is the range between the three factors of \(k_1\), \(k_2\), and \(k_3\). The results are shown in table 5.

| \(k_1\) | \(k_2\) | \(k_3\) | \(R\) |
|---------|---------|---------|-------|
| 25.513  | 11.824  | 6.646   | 18.867|
| 14.824  | 15.43   | 13.729  | 1.701 |
| 15.345  | 14.062  | 14.576  | 1.283 |
| 14.967  | 14.504  | 14.512  | 0.455 |

From table 5, \(R_A>R_B>R_C>R_D\), the effect of CNTs solution concentration is most significant, vibration frequency and drying temperature are the second, and the influence of dip dyeing time is the smallest. The electrical performance of the heating wire is inversely proportional to the resistance. The smaller the resistance, the better the electrical performance. Therefore, the smaller the \(k\) value, the better the process parameters. The optimal combination of each factor in table 5 is \(A_3B_3C_2D_2\), involving the vibration frequency of 80 Hz, the drying temperature of 40°C, and the dip dyeing time of 2 min. On this basis, the single factor of the concentration of carbon nanotube solution was studied in depth.

To further clarify the influence of the solution concentration on the forming effect of the heating wire, the carbon nanotube solution concentration (2 wt%, 4 wt%, 6 wt%, 8 wt%, 10 wt%) is changed to perform the heating wire forming test under the same conditions of other processes. The average value and standard deviation of the heating wire resistance of 10 cm in length are selected as test indicators,
and the measurement results are shown in figure 2.

![Figure 2. The influence of different concentrations of CNTs solution on the resistance of heating wires.](image)

From figure 2, as the concentration of the CNTs solution increases, the resistance of the heating wire decreases first and then increases. When the concentration of the CNTs solution is 8 wt%, the average value and standard deviation of the heating wire resistance are the smallest. This is because when the concentration of the solution is low, the amount of printing of carbon nanotubes on the polyester yarn is small, the conductive path formed is small, which causes the result in a large resistance of the heating wire. As the concentration of the CNTs solution increases, the viscosity of the solution increases, the fluidity decreases, so that the content of carbon nanotubes between the polyester fibers decreases and the resistance of the heating wire becomes larger.

### 4.2. Polymer content

To improve the wear resistance and washing resistance of the heating wire, according to the formulation of table 6, two polymers (waterborne polyurethane and nitrile latex) are added to the carbon nanotube solution in a ratio of 1:1, stirred, vibrated by ultrasonic for 1 hour. The heating wire forming test is carried out under the condition that other process conditions are unchanged. The average value and standard deviation of the heating wire resistance of 10 cm in length are selected as test indicators, and the measurement results are shown in table 6.

| Sample number | Polymer content (g/L) | Resistance (kΩ) |
|---------------|------------------------|-----------------|
| S1            | 2.5                    | 2.406±0.49379   |
| S2            | 5                      | 3.053±0.28203   |
| S3            | 7.5                    | 36.073±6.02204  |
| S4            | 10                     | 262.133±39.8278 |
| S5            | 12.5                   | 5.787±0.77125   |
| S6            | 15                     | 1.805±0.19081   |
| S7            | 17.5                   | 1.815±0.25558   |
| S8            | 20                     | 177.18±10.72477 |

From table 6, as the polymer content increases, the resistance of the heating wire shows a tendency to increase first, then decrease and next increase. When the polymer content is increased from 2.5 g/L to 15 g/L, the resistance of the heating wire is first increase and then decrease; after the polymer content...
exceeds 15 g/L, the resistance of the heating wire begins to increase again. Therefore, when the polymer content is 15 g/L, the average value and standard deviation of the heating wire resistance are small, and the resistance reaches a minimum value of 180.5 Ω/cm.

To further observe the morphology of the heating wire by scanning electron microscopy, which can observe the microstructure of the heating wire of S1 group and S6 group. Figure 3 is a micro-morphology of the heating wire, the magnification of a and c is 6000, and the magnification of b and d is 30,000.

![Figure 3. Micro-morphology of heating filament.](image)

(a) SEM of S1 at low power;(b) SEM of S1 at high power(c) SEM of S6 at low power;(d) SEM of S6 at high power.

From the low-power electron micrograph of figures 3(a) and 3(c), a layer of carbon nanotubes is evenly distributed on the surface of the heating wires of groups a and c, without obvious protrusions and breaks. The surface of the heating wire of group c is more dense and smooth. From the high-power electron micrograph of figures 3(b) and 3(d), the carbon nanotubes on the surface of the heating filaments of group b are arranged intricately to form a conductive path. However, the carbon nanotubes are attached to the surface of the polyester fiber in a monomer form. It is easy to fall off when rubbed or washed. The surface of the heating wire of group d has a conductive film composed of polymer and carbon nanotubes, the carbon nanotubes are evenly filled in the polymer. While maintaining the original electrical conductivity, the wear resistance and washing resistance of the heating wire are improved.

4.3. Heating test

To measure the heating performance of the heating wire, 15 heating wires of S6 group are horizontally arranged into a heat-generating sample with an effective heating area of 5×8 cm², the ends of the heating wire are short-circuited with a conductive copper foil tape to form a parallel structure, the resistance after paralleling is about 170.4 Ω. The heat-generating sample is connected to a DC voltage of 12 V; an electronic thermometer is used to collect the surface temperature of the heat-generating sample after the energization, and the temperature change curve with time is shown in figure 4.

With the extension of the energization time, the surface temperature of the heat-generating sample gradually increases, and the growth rate gradually decreases from figure 4. When the power-on time is greater than 600 seconds, the surface temperature no longer increases and the maximum heating temperature is 52.5°C. It is indicated that the prepared heating wire can be heated at a low pressure, which lays a foundation for the preparation of the subsequent low-pressure heating fabric.
4.4. The test of washing resistance

To test the washing resistance of the flexible heating wire, the S6 group of flexible heating wire with a length of 10 cm is placed in water, under normal temperature conditions, with a magnetic stirrer (speed to 1500 rad/min) mechanical agitation for 3 minutes and then dried at 40°C. The treatment is repeated 10 times, and the resistance of the heating wire after each water washing is measured with a digital multimeter. The results are shown in figure 5.

From figure 5, the resistance of the heating wire changed greatly after the initial washing and fluctuated slightly after washing. The main reason is that the initial washing causes bending of the heating wire in many places, which causes increase of the resistance value of the heating wire. After the initial washing of the heating wire, the resistance of the heating wire is not greatly changed due to the washing times, which proves that the heating wire has a fair washing resistance and lays a foundation for the preparation of the subsequent water-resistant heating fabric.

5. Conclusions

- The optimal printing and dyeing process parameters are obtained through the flexible heating wire forming system, multi-factor orthogonal analysis and single factor test. When the carbon nanotube solution concentration is 8 wt%, the vibration frequency is 80 Hz, the drying temperature is 40°C, the dip dyeing time is 2 min and the polymer content is 15 g/L, the average value and standard deviation of the heating wire resistance are smallest, and the resistance reaches a minimum value of 180.5 Ω/cm.
- The microscopic morphology of the heating wire shows that a layer of carbon nanotube monomer is uniformly distributed on the surface of the heating wire without obvious protrusion and fracture. The carbon nanotubes on the surface of the heating wire of the S1 group are attached in a monomer form, and the S6 group are uniformly filled in the polymer to form a conductive film.
- The heating property and washing resistance of heating wire were tested. The test shows that with the extension of the energization time, the surface temperature of the heating sample gradually increased, and the highest heating temperature was 52.5°C; the resistance of the heating wire changed greatly after the initial washing and fluctuates slightly after washing.

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