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Fabrication of High Nano-ZnO Doped with Boric Acid Assembled on Cotton Fabric with Flame Retardant Properties

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Abstract
In this study, an attempt was made to investigate the flame retardancy of cotton fabrics coated with a high nano-ZnO content. Via a simply method in situ, a novel ZnO/cotton composite can be fabricated with a high proportion of nano-ZnO assembled on cotton fabric, where the finished cotton fabric has a certain property of flame retardancy. Discussed herein is the effect of the processed liquid concentration, ammonia-smoking time, and curing temperature on fabric properties such as flame retardancy and hand feeling. Performed were also tests of doping boric acid using the vertical burning method. The finished cotton fabrics were analysed using X-Ray Diffrcation (XRD) and a field emission scanning electron microscope (FESEM), which indicated that between the fibres, and inside the lumen and mesopores of the cotton fibres are assembled with nano-ZnO. The results show that the nano-ZnO content on cotton fabrics can reach up to 15.63 wt%, with the finished cotton fabric having excellent flame retardancy, despite the long after-glow time; however, doping with 0.8 wt% boric acid on the cotton fabric can markedly reduce this. Therefore, a high amount of nano-ZnO doped with boric acid assembled on cotton fabric has great potential in the future.

Key words: *high-load, ZnO; boric acid, cotton fabric, flame retardant.*

Introduction

Cotton fibre has a wide range of applications in military and civilian areas, such as apparel and home furnishings, because of the comfort and breathability of garments made of it [1]. However, the high flammability of cotton presents a significant threat to humans and their possessions [2]. A number of serious burning injuries associated with cotton fabrics occur annually worldwide [3]. Conferring flame retardancy to cotton fabrics can reduce these occurrences and contribute to saving lives and properties. Therefore, it is urgent to improve the fire behaviour of cotton [4-6].

The flame retardancy of cotton fabric can be achieved by chemical treatment, and halogen-based and formaldehyde-based flame retardants have shown remarkable flame retardancy in the past decades [7, 8]. However, formaldehyde is recognised as a carcinogenic compound by the World Health Organisation and halogen-containing compounds in combustion will release hydrogen halide toxic and corrosive gases [9]. Therefore, halogen-based chemicals are strictly banned in the market [10].

In recent years, nitrogen-phosphorous flame retardant synergism has received considerable attention because it provides excellent fire protection [11]. A compound containing phosphorous and nitrogen could achieve effective flame retardancy by the synergistic action [12, 13], but it is usually confronted with problems of water resistance [14]. Moreover, the treatment process above is not eco-friendly, and a large quantity of costly synthetic chemicals are consumed. Hence, there is an increasing demand for inexpensive, eco-friendly fire retardant agents. In this direction, researchers have invented various plant and protein-based bio-macromolecules, such as DNA, for making sustainable fire retardant textiles [15, 16].

Nano-ZnO is non-toxic, eco-friendly and harmless to humans [17]. Some research has been done based on its antibacterial and UV-blocking properties [18-20]. However, there are few researches about flame retardancy. Kumanan Bharathi Yazhini found that nano-ZnO is effective in reducing the flammability of finished fabrics [21]. In this paper, while the ZnO content assembled on cotton fabric reached a certain degree, the flame retardancy of the finished cotton fabric should be improved greatly, classified as condensed phase flame retardancy. The specific mechanism may be a large amount of the inorganic filler ZnO in flame retardant materials. However, packing can dilute the flame retardancy of combustible material, and ZnO has a large heat capacity and heat storage, with high thermal stability. Thus, in cotton fabric finished with flame retardant material, it is not easy to achieve a thermal decomposition temperature. Boric acid is also environmentally-friendly and can be used as

a flame retardant. Antoni Niekraszewicz found boric acid in anti-mite fabric as having a positive effect on the well-being of patients with a mite allergy [22]. Kongliang Xie synthesised compounds containing boron and nitrogen-based boric acid, which were used in flame retardant cotton fabric [23].

This study aimed at adopting a method different from traditional finishing, using pre-treated cotton fabric, zinc acetate and ammonia as raw materials to fabricate high nano-ZnO assembled cotton fabric via a method in situ. Then the ZnO/cotton composite was finished with boric acid. This paper mainly studied the preparation process and related factors in relation to the properties of the cotton fabric after finishing, especially the flame retardancy.

Experimental

Materials

Bleached cotton fabric, whose warp and/weft density was 455 × 265 root/10 cm, 30 wt% hydrogen peroxide solution(H_2O_2), sodium hydroxide(NaOH), urea(CON_2H_4), zinci acetas dihydricus($Zn(CH_3COO)_2 \cdot 2H_2O$), ammonia($NH_3 \cdot H_2O$), ice acetic acid($CH_3COOH$), and boric acid($H_3BO_3$) were of analytical grade and used without any further purification, purchased from the Linfeng Chemical Company of Shanghai. Cotton fibres were obtained from a commercial market.

Treatment process

Preparation of cotton fabric

In order to oxidate cotton fibres weakly at a constant temperature of 40 °C, the original cotton fabrics were immersed in 30 wt% hydrogen peroxide solution for 30 min (bath ratio 1:30). After the treatment, the cotton fabrics were rinsed with clear water up to neutral conditions and dried using a drying oven at 80 °C.

The cotton fabrics were completely immersed in the mixture of the solution centaining 9 wt% sodium hydroxide and 9 wt% urea at constant temperature of 5 °C with low tension for 12 hours. After the treatment, the cotton fabrics were rinsed with clear water up to neutral conditions (pH = 7).

Fabrication of nano-ZnO assembled cotton fabrics

The fabrication of ZnO-assembled cotton fabrics was carried out as follows: Firstly, zinci acetas dihydricus was dissolved in distilled water to make a zinc ion concentrations of 5 wt%,10 wt%, 15 wt% & 20 wt%, respectively, and then the alkali-treated cotton fabrics were completely immersed in zinc acetate solution for 10 min. When the concentration of zinc ion was higher than 15 wt%, through the method of increasing the solution concentration gradually by immersing the fabrics, from 15 wt% to 20 wt%, the fabrics were then placed in a lidded beaker with strong aqua. There was a bracket used to support the cotton fabric placed in the beaker, in which there was a suitable temperature to accelerate volatilization of the concentrated ammonia. Then the ammonia-smoking cotton fabrics were taken out after a certain time, and then put in a ventilated place for 30 min. Finally, the cotton fabrics were dried at 80 °C for 5 min, cured for 2 min at 150 °C, then washed with hot water, and dried at 80 °C.

Doping with boric acid

ZnO/cotton composite fabric was impregnated with different concentrations of boric acid in the solution, dried with padder after being taken out, and then dried at 100 °C.

Measurements

Water absorption

A cotton fabric sample of 10 cm × 10 cm size was placed in a dry container to balance for 24 h before weighing G_0, then put into a certain amount of distilled water (bath ratio 1:100) for 1h before taking out, and then G_1 was weighed after the excess liquid was removed. The water absorption of the fabric was calculated (g water/g fabric), according to the formula:

$$A = (G_1 - G_0)/G_0 \quad (1)$$

XRD

The samples obtained were characterised using an X-ray diffractometer (XRD) – D/max-2550VB+PC, equipped with a CuKa radiation tube operating at 40 kv and 30 mA at room temperature. The speed of canning was 5.0 (°)/min and diffractograms were obtained at scattering angles from 10° to 80°.

FESEM

Surface morphologies of the samples were observed by field emission scanning electron microscopy (FESEM). The samples were platinum-coated using a sputter coater in order to capture clear images of the surfaces.

Vertical buming test

Flame retardant properties of the nano-ZnO assembled cotton fabrics were investigated by means of a YG815B type vertical buming test instrument to test the vertical combustion property of the finished cotton fabrics, on accordance with GB/T 5455-1997. Under the specified test conditions, the following test indexes: after-flame time (s), after-glow time (s) and the char length (mm) were used to evaluate the flame retardant properties of the flame retardant textiles [24].

Handle

The handle of the cotton fabric was assessed using the subjective evaluation method. Pinching, touching, grasping and looking were used to evaluate the handlé characteristics of the fabric, such as smoothness, softness, fullness, elasticity, firmness, body bone and flexibility. For a more intuitive comparison, a five-level representation was used, with the worst being level 1 and level 5 the best; Unfinished fabric is level 5.

Content of nano-ZnO and boric acid

The content of zinc assembled on cotton fabric is defined in terms of nano-ZnO, which is measured by the weighing method. An electronic balance was used for a 10 cm × 10 cm size of the unassembled and assembled cotton fabrics, G and G' respectively. According to the following formula, the content, C, was calculated.

$$C = (G' - G)/G' \times 100\% \quad (2)$$

Electronic scales were to weigh ZnO/cotton fabric G_1, the fabric impregnated with boric acid solution G2, and G3 after drying. The concentration of boric acid as a % was calculated according to the following formula, represented by D.

$$D = [(G_2 - G_1) \times a\%]/G_3 \times 100\% \quad (3)$$

Washing durability test

A washing durability test was conducted according to GB/T 3921.3 1997 with 5 g/l of a neutral detergent solution at a bath ratio of 1:50, washing for 10 min at 40 °C as a cycle, then rinsing off with clear water, washing a certain number of times repeatedly, and then drying at 80 °C [25].

Results and discussion

Swelling pretreatment analysis

When the cotton fabric was immersed in distilled water, water molecules went

inside pores fibreand between fibres. The larger the internal porosity of the cotton fibres fibre, the more water molecules went into the pores. Thus, the water absorption of cotton fabrics can be reflected in the size of the pore capacity of cotton fibres after swelling. The inside pores of fibres can hold more nano-ZnO particles, and Fibreif the internal porosity of fibres become larger, the more nano-ZnO will be assembled in cotton fabrics. The unswelled and swelled cotton fabrics were measured for their water absorption (g water/g fabric), which was 2.87 g water/g fabric and 4.55 g water/g fabric, respectively. It can be seen that the water absorbption capacity of cotton fibres obviously increased after the swelling treatment. The reason may be the occurrence of swelling after the alkali treatment, and the oxidation of cellulose fibre leads to the internal lateral connections between macromolecular chains being weakened, and to an increase in the structure of cellulose fibres in the amorphous region. The increased amorphous region provided more places for adsorption, resulting in a significant increase in the water absorbing capacity, which indicated that the inside pores of fibresfibre had obviously increased in the aqueous solution, and can hold morefibre nano-ZnO particles.

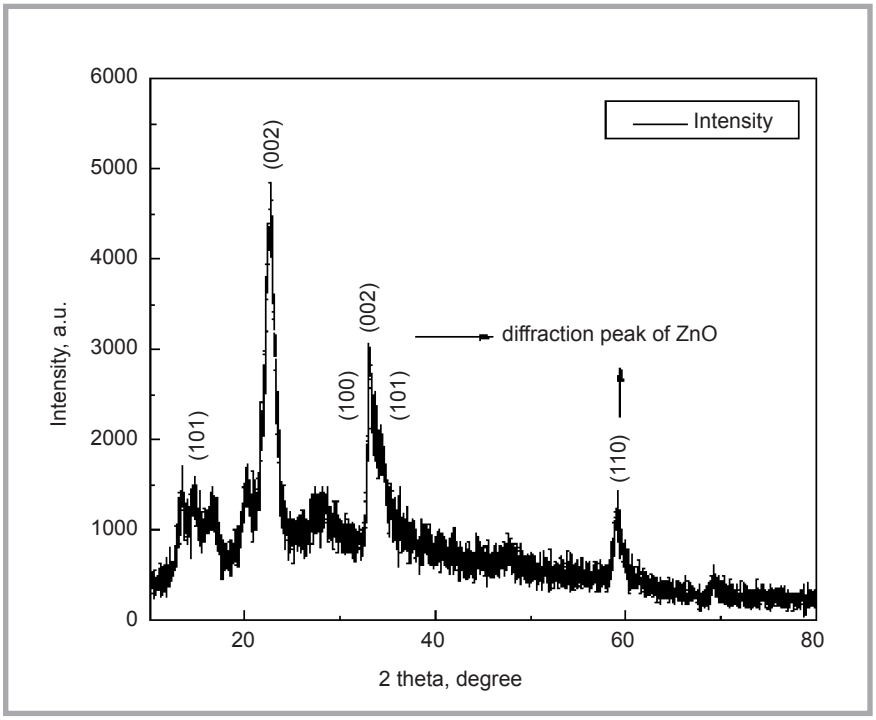

Figure 1. *XRD patterns of high nano-ZnO assembled cotton fabric.*

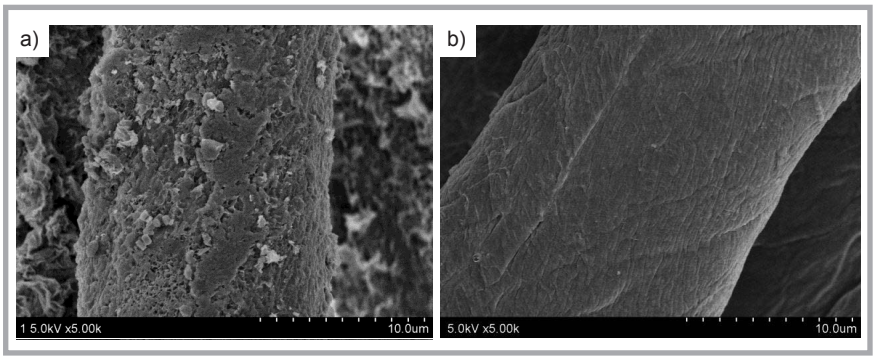

Figure 2. *SEM images of cotton fibres: a) ZnO assembled cotton fibres, b) ZnO assembled fibres after washing 30 times.*

Table 1. *Relationship between zinc ion concentration of treated liquid and property of flame retardancy.* ***Note:*** *Ammonia-smoking time – 10 min, curing temperature – 150 °C.*

Zinc ion concentration of treated liquid, %	After-flame time, s	After-glow time, s	Char length, cm	Content of ZnO, %	Handle level
0	19.7	31.1	30.0	0	5
5	17.6	18.8	30.0	6.54	5
10	10.2	10.5	30.0	9.96	5
15	0	54.6	5.9	15.63	4
20	0	33.6	3.5	18.44	3

XRD analysis

X-ray diffraction (XRD) was used to observe the crystallinity and crystal phase of the treated cotton fibres assembled with ZnO nanoparticles. 15.63 wt% nano-ZnO assembled in cotton fabric is shown in ***Figure 1***. The peaks at (101), (002) are the diffraction peaks of the cotton fibre (cellulose I) , which indicates that itfibre exhibited a cellulose I structure. In addition to the diffraction peaks of the cotton fibre (cellulose I),there are also strong diffraction peaks in the curve at 2theta values of 33.18°, 33.74°, 34.32° & 59.24°, corresponding to the (100), (002), (101) and (110) planes, respectively, and can readily be indexed to the phase of ZnO. The peaks corresponding to the nano-ZnO (100), (002), (101) and (110) planes are distinct, proving that a great amount of nano-ZnO deposition was generated on the cotton fabrics, which have a high ZnO content. The crystallite size of ZnO, calculated using Scherrer's formula, was found to be 19.18 nm.

FESEM analysis

In order to observe the distribution of the in situ deposition of ZnO on cotton fibres, FESEM was used to observe the surface morphology of the treated cotton fibres and treated fibres after washing 30 cycles, ***Figures 2.a*** and ***2.b*** show images of the treated cotton fibres.

As shown in ***Figure 2.a***, there was a large number of particles on the surface of the cotton fibres. As shown in ***Figure 2.b***, the surface of the cotton fibre is smooth, with no attachments, and it shows that ZnO on the surface of the cotton fibre had been washed away after washing 30 cycles. It also reveals that adhesion of ZnO deposition on the surface of fibres is not enough. The existence of ZnO and its content in the fabrics will be discussed in the following washing durability test section, detailing FESEM observation of just the surface morphology of fibres.

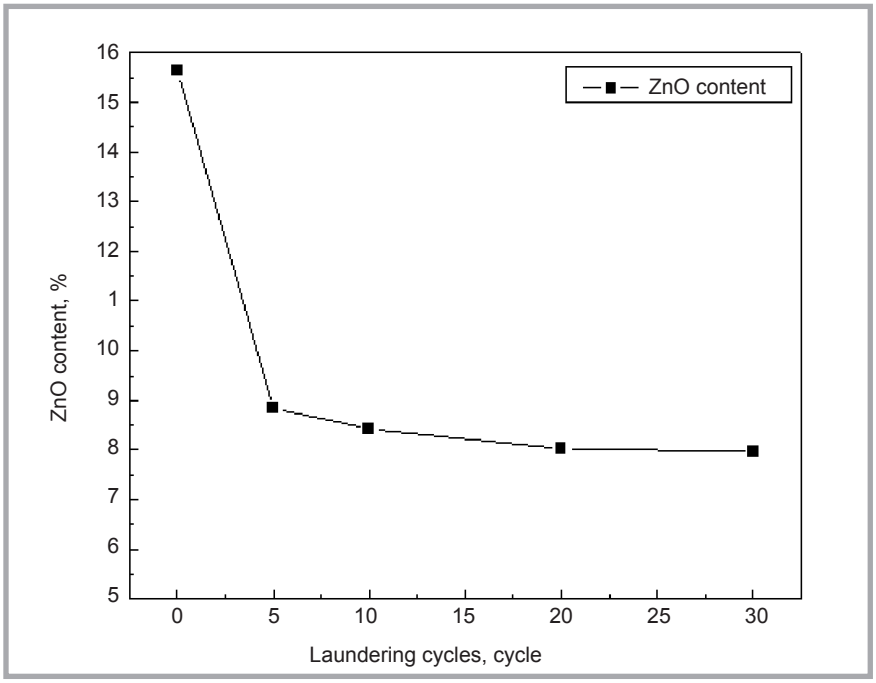

Figure 3. *Curve of laundering cycles and ZnO content.*

Table 2. *Relationship between ammonia-smoking time and flame retardancy property.* ***Note:*** *Zinc ion concentration of processed liquid – 15 wt%, curing temperature – 150 °C.*

Ammonia-smoking time, min	After-flame time, s	After-glow time, s	Char length, cm	Content of ZnO, %	Handle level
5	9.8	12.0	30.0	10.42	4
10	0	54.6	5.9	15.63	4
15	0	53.2	6.0	15.88	4

Table 3. *Relationship between curing temperature and property of flame retardancy.* ***Note:*** *Zinc ion concentration of processed liquid – 15 wt%, ammonia-smoking time – 10 min.*

Curing temperature, min	After-flame time, s	After-glow time, s	Char length, cm	Content of ZnO, %	Handle level
120	0	56.2	6.1	15.63	4
135	0	55.1	6.0	15.63	4
150	0	54.6	5.9	15.63	4
165	0	54.5	6.0	15.63	3

Evaluation of flame retardancy

Influence of zinc ion concentration on the flame retardant property

The relationship between the zinc ion concentration of treated liquid and the property of flame retardancy is shown as ***Table 1***.

It can be seen from ***Table 1*** that with an increase in liquid containing zinc ions, the after-flame time and char length of the cotton fabrics became shorter; thus, the the cotton fabrics' flame retardancy had been improved significantly. With a higher concentration of processed liquid of zinc ions on the cotton fabric, the amount of nano-ZnO will be more in the end. When the liquid zinc ion concentration in the cotton fabrics increased from 0 to 10 wt%, the handle level of the cotton fabrics did not change, and properties of flame retardancy increased indistinctly. After burning, the cotton fabrics were completely damaged, and thus the after-glow time was shorter than for a 10-15 wt% zinc ion concentration of the treated fluid. When the zinc ion concentration increased from 15 to 20 wt%, there was a clear improvement in the high nano-ZnO assembled cotton fabric's flame retardancy was. However, when the zinc ion concentration reached up to 20 wt%, the fabric comfort level dropped two levels, and the strength loss was higher. Hence, the zinc ion concentration in the liquid should not be too high, and it is advisable to choose a 15 wt% concentration of zinc ion treated fluid.

Influence of ammonia-smoking time on the property of flame retardancy

The relationship between the ammonia-smoking time and flame retardancy property is shown in ***Table 2***.

Table 2 shows that with an increase in the ammonia-smoking time, the after-flame time and char length of the cotton fabrics became shorter; thus, the cotton fabric's flame retardancy performance was also improved. As the ammonia-smoking time became longer, the strong aqua ammonia, which volatilized come into contact with the treatment fluid on the cotton fabrics, produced by the more complete reaction of hydroxyl ions and zinc ions and by more particles of nano-ZnO generated at the time of curing. When the ammonia-smoking time was more than 10 min, the cotton fabrics containing zinc ions in the processed liquid underwent an almost complete reaction, and if ammonia-smoking was continued, the content of nano-ZnO in the cotton fabrics was nearly unchanged; hence, the smoldering time and damaged length were also nearly unchanged. Regarding saving raw materials and the experimental time, it is advisable to choose 10 min as the ammonia-smoking time.

Influence of curing temperature on the property of flame retardancy

The relationship between curing temperature and flame retardancy is shown in ***Table 3***.

It can be seen from ***Table 3*** that with an improvement in the curing temperature, the after-glow time and char length were nearly unchanged, hence, the flame retardancy properties of the cotton fabrics were almost unchanged. It shows that there was almost no relationship between the cotton fabric's flame retardancy properties and the curing temperature. Within certain limits of temperature, elevated temperature is advantageous to nano-ZnO particle cross-linking with the reactive group of the cotton fabric. Therefore, the cotton fabric is finished with a certain amount of flame retardancy. With a curing time of 2 min and with an increase in the curing temperature, the nanometer zinc oxide and cotton fabric were cross-linked; however, after finishing, the cotton fabric's after-flame time, after-glow time and char length were almost unchanged. When the temperature is higher than 150 °C, the finishing of cotton fabrics becomes

yellow, the feel hardened, and the fracture strength loss rate and char length increase. When the temperature is below 150 °C, the degree of the crosslinking reaction is reduced, and then the ZnO-assembled on the cotton fabrics is easy to be washed away. Above all, it is advisable to choose a baking temperature at 150 °C.

Influence of contents of doped boric acid on the property of flame retardancy

The relationship between contents of doped boric acid and the property of flame retardancy is shown in ***Table 4***.

It can be seen from ***Table 4*** that along with an increase in the boric acid content from 0 to 0.8 wt%, the after-glow time decreased greatly, as well as the char length of the fabric. According to GB 20286-2006 [26], "flame retardant products in public places and component combustion property requirements and identity" (flame retardant grade 1: char length ≤ 150 mm, after-flame time ≤ 5 s, after-glow time ≤ 5 s; flame retardant grade 2: char length ≤ 200 mm, after-flame time ≤ 10 s, after-glow time ≤ 10 s), the fabrics meet the requirements of flame retardant grade 1. After the boric acid content increased from 0.8 wt% to 1.2 wt%, the after-glow time and char length were also clearly unchanged, which meant that at this time a small increase in boric acid has no effect on the flame retardancy properties of cotton fabric.

Washing durability analysis

To select the best process conditions of cotton fabric finishing, analysis of the influence of laundering cycles on the properties of ZnO-assembled on cotton fabric with respect to flame retardancy was made. The laundering frequency in relation to the ZnO-assembled curve is shown in ***Figure 3***, from which it can be seen that with the increase of laundering cycles, nano-ZnO content in the cotton fabrics was reduced gradually. The loss rate of 5, 10, 20 and 30 laundering cycles was 43.38 wt%, 48.42 wt%, 52.87 wt% and 48.42 wt%, respectively, indicating that after 30 laundering cycles, the content of ZnO in cotton fabrics remains basically the same; which means that with 15.63 wt% ZnO in the presence of almost half of the cotton fibres' internal pores and lumen, the content of ZnO is still high after 30 laundering cycles.

Table 4. *Relationship between contents of doped boric acid and property of flame retardant.* ***Note:*** *Zinc ion concentration of processed liquid –15 wt%, ammonia-smoking time – 10 min, curing temperature – 150 °C.*

Contents of boric acid doped, %	After-flame time, s	After-glow time, s	Char length, cm	Content of ZnO, %	Handle level
0	0	54.6	5.9	15.63	4
0.4	0	22.6	3.2	15.63	4
0.8	0	1.2	2.1	15.63	4
1.2	0	1.0	2.0	15.63	3

Table 5. *Relationship between the content of doped boric acid and property of flame retardancy.* ***Note:*** *Zinc ion concentration of processed liquid – 15 wt%, ammonia-smoking time – 10 min, curing temperature – 150 °C.*

Laundering cycles, cycle	After-flame time, s	After-glow time, s	Char length, cm	Content of ZnO, %	Handle level
0	0	54.6	5.9	15.63	4
5	10.4	6.5	30.0	8.85	5
10	12.7	8.9	30.0	8.42	5
20	13.4	10.1	30.0	8.03	5
30	14.0	12.3	30.0	7.97	5

The relationship between the content of doped boric acid and the property of flame retardancy is shown in ***Table 5***.

Table 5 shows that at a high nanometer zinc oxide content, the cotton fabric has excellent flame retardancy. Along with the increase of laundering cycles, the after-flame time and after-glow time of the cotton fabrics increased; the fabrics were damaged completely, and their flame retardant property was not obvious. When the laundering cycles were risen to 30 times, the flame retardant property of the cotton fabric remained basically unchanged.

Conclusions

It is concluded that the optimal assembly for the process through experiment is as follows: zinc ion concentration of processed liquid – 15 wt%, ammonia-smoking time – 10 min, curing temperature – 150 °C, and content – up to 15.63 wt% of nano-ZnO in high nano-ZnO assembled fabric. High nano-ZnO assembled cotton fabrics possess an excellent flame retardant property, and they are harmless to the environment and human body; but the after-glow time is too long, and thus it cannot be used as a flame retardant material. After doping with boric acid, the after-glow time of the finished fabric clearly decreased. Cotton fabric will have excellent flame retardancy if the nano-ZnO content is more than 15.63 wt%, and when the boric acid content is 0.8 wt%. Therefore, high nano-ZnO doped with boric acid assembled on cotton fabric has great potential for the future.

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