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Preparation of Nano-ZnO and Its Application to the Textile on Antistatic Finishing

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Abstract

Nano-ZnO was prepared by direct precipitation method with zinc chloride and sodium carbonate anhydrous as raw materials, and its particle size and dispersion were characterized by TEM. The effects of concentration and the ratio of reactants and reaction temperature on its dispersion in aqueous solution were analyzed and the best reaction conditions were as follows: reaction temperature 60℃, ultrasonic vibration time 40 min, concentration of the reactant 0.5mol/l and molar ratio of reactants 1:2 for the preparation. The cotton fabric and the polyester fabric which were both finished by pad-dry-cure process with antistatic finishing agent, which was compounded with nano-ZnO, were tested on their antistatic property. According to the comparation between the cotton and the polyester treated fabrics on antistatic property, the results showed that the charge density of the polyester fabric was reduced to 9.5×10^{-8} C/m^2 from 5.8×10^{-6} C/m^2, which was about 10 times as that of the cotton fabric.

Keywords: Nano-ZnO, Direct precipitation method, Cotton fabric, Polyester fabric, Antistatic finishing

1. Introduction

To protect human health and improve human living environment, many researchers have been paid attention to functional textiles in recent years (Hee, 2007, p.8020). And the rapidly developing nanotechnology become a research hotspot, because the special physical and chemical properties, which are beneficial to study on the functional products and produce many new applications, are showed when the bulk particle size is reduced to nanometer range (Herrera, 2006, p.245; Iijima, 1999, p.297; Kruis, 2001, p.39). In the metal oxides that are widely used in different areas, zinc oxide powders are important material for applications due to their unique optical, electrical, dermatologicaland antibacterial properties (Takahashi, 2007, p.722; Alessio, 2007).Currently, the synthesis approaches of nano-ZnO are various, which are mainly divided into three kinds: the solid, the solution and the vapor phase methods (Muccillo, 2004, p.302).In this paper, nano-ZnO were obtained through a direct precipitation reaction, which belongs to the solution phase methods, between zinc chloride and sodium carbonate anhydrous. The effects of reaction temperature, concentration and the ratio of reactants, and so on, on the deposition of nano-ZnO in aqueous solution were discussed. And nano-ZnO was used to prepare the nanometer antistatic finishing agent, which was applied to treat cotton and polyester fabrics, and then the antistatic performance of the treated textiles was evaluated through the charge density (Alessio, 2007).

2. Experimental

2.1 Synthesis of nano-ZnO

Zinc chloride($ZnCl_2$, purity 98.0%), sodium carbonate anhydrous(Na_2CO_3, purity 99.8%) and Ethanol absolute(CH_3CH_2OH, purity 99.7%) were obtained from Tianjin Chemical Reagent Ⅲ Co. Zinc chloride was of chemic grade, and all the other reagents were of analytical grade. surfactant and 106 binder were purchased from market. All were used as received without any further purification in the experiment.

The preparation of nano-ZnO according to the literature (Liao, 2003, pp.22-23): the nano-ZnO was prepared by direct precipitation method, $ZnCl_2$ and Na_2CO_3 were dissolved in water respectively. And then the Na_2CO_3 solution was dropped into $ZnCl_2$ solution, and that resulted in precipitation, then it was treated with ultrasonic vibration and aged several hours. In order to remove salt, as-burnt powders were filtered then rinsed in distilled water and ethanol. Finally, the precursor was dried in oven at 80℃ for 4 h, and thermal treatment at 450℃ for 3 h led to the formation of

nano-ZnO (Chen, 2006, p.435).

2.2 Fabric treatments

White cotton and polyester fabrics were used as received. The mass per unit surface was 104 g/m^2 for cotton and 150 g/m^2 for polyester (Alessio, 2007).

A certain concentration of nanometer antistatic finishing agent was prepared as follows: a certain amount of nano-ZnO and surfactant were added into distilled water and the mixture was mixed well with ultrasonic vibration for several minutes, and then the adhesion agent is added under stirring (Li, 2006, p.265).

The cotton and polyester samples were soaked for 5 min in the nanometer antistatic finishing agent, then the clothes were squeezed to remove the excess finishing agent by a NM—450 open-width impregnator (two dipping androlling).The treated fabrics were dried and baked in a DK—5E curing machine, respectively, at 80℃for 1 min and 160℃ for 3min (Alessio, 2007).

2.3 Measurements

The morphologies and size of the products were characterized by a Hitachi Model H-7650 transmission electron microscope (TEM) (Tang, 2006, p.548). The charge density of the fabrics with and without finishing were measured according to a standard method (Professional Standards of the People's Republic of China ZB W 04008-89). The whiteness of the fabrics with and without finishing were performed via a WSD—3U fluorescent whiteness meter.

3. Results and discussion

3.1 The impact of reaction conditions

The size and quality of the particles have effect on particle's settling velocity in dispersion medium. The larger the particle, the higher settling velocity it is (Ni, 2006). Nanoparticles owe to the properties of small dimension, large specific surface area and high surface activity, which lead to agglomeration easily (Luo, 2003, p.27). Settlement experiment was employed to estimate the diameter and dispersion in aqueous solution of nano-ZnO through the deposition time. The results show that the deposition time is longer, the particle is smaller and the dispersion is better (Cui, 2001, p.99). In this experiment, various experimental conditions were analyzed based on the deposition time of nano-ZnO in aqueous solution.

3.1.1 The impact of time of the ultrasonic vibration

Mechanical action caused by ultrasonic vibration within the submicroscopic range including mechanical mass transfer, heating effect and cavitation effect, in which, the pulverization effect produced by transient cavitation effect make the precipitation becoming homogeneous minimal particles and cavitation bubbles, which reduce the specific surface free energy of crystal nucleus, formed by steady cavitation effect inhibit the agglomeration and growth of the crystal nucleus, so the particle size of the product is reduced(Liang, 2002, p.570; Qiu, 1999, p.45; Zhang, 2002, p.82). However, heating effect will raise medium temperature, and that increases the collision probability of the particles. Which lead to a reagglomeration of the dispersed particles, forming a second-level agglomeration, so speed up the settlement of the particles and result in a declined dispersion (Qiu, 1999, p.45; Jiang, 2007, p.725; Luo, 2007, p.185).As can be seen from Fig. 1, with the increase of time of the ultrasonic vibration, the deposition time of nano-ZnO in aqueous solution increased at first and then decreased. This is because the increase of time of the ultrasonic vibration leads to fully cavitation effect, and heating effect become more and more obviously when the time went on continually. Therefore, the time of ultrasonic vibration of 40 min was chosen for experiment.

3.1.2 The impact of temperature

The deposition time of nano-ZnO in aqueous solution present a trend of first increases then decrease, which is shown in Fig. 2. This is due to the temperature has an impact on supersaturation of solution, which relate with the nucleation rate in system. As the temperature rises, the faster reaction rate and the increasing of supersaturation of reaction products lead to crystal core forming rate was accelerated in the short reaction time, that means the controlling step of the reaction is transferred from grain growth to crystal nucleus formation, so the particles are smaller. With the temperatures continuing to rise, the phenomenon of "nuclear-aggregation" caused by the rapid formation of crystal nucleus is obvious that result in forming aggregate among the crystal nucleus (Wang, 2006, p.41; Kang, 2005, p.348). Therefore, 60 ℃ was the best temperature.

3.1.3 The impact of concentration of zinc chloride

As shown in Fig. 3, the concentration of reaction has a great effect on products' diameter. With the increase of the supersaturation of the solution in response to the increasing of ion concentration in system, a great deal crystal nucleus are generated instant of the reaction, which lead to tiny crystalline grains increased. So the "the size fraction agglomeration" phenomenon becomes gradually significant, and that result in the larger particles (Kang, 2005, p.348; Wang, 2006, p.41; Ding, 2002, p.1016). The deposition time of the nano-ZnO in aqueous solution show a trend of

decreasing, which reflect a change of diameter of the products. The dispersion of nano-ZnO in aqueous solution was the best when the concentration of $ZnCl_2$ was 0.5mol/l.

3.1.4 The impact of ratio of the reactant

The deposition time of nano-ZnO which was prepared with different ratios in aqueous solution are 2-6 h, the results indicate that the impact of the ratio of reaction on products' dispersion is not obvious. As is shown in Fig. 3, the ratio of the reaction has a greater impact on production of precursor. This is because the nanoparticles were prepared by direct precipitation method, which use the precipitation reaction among reactants, and the precipitation-dissolution equilibrium exists in reaction system, whose direction is influenced by the common ion effect and the salt effect. With the increasing of the reactant content, the leading role of the common ion effect and the salt effect, respectively, gradually weaken and enhance. In order to ensure the precipitation-dissolution equilibrium, the directions of reaction change from deposit generation to dissolution (General chemical staff room of Tianjin University, 1983), which lead to a trend of the precursor output that first increased then decreased.

Considering the effects of ratio of the reactant on the dispersion of nano-ZnO in aqueous solution and precursor output, the best molar ratio of zinc chloride and sodium carbonate anhydrous was 1:2.

3.2 TEM analysis of nano-ZnO

In the process of nano-ZnO preparation by direct precipitation method, the stirring, reaction temperature, concentration and the ratio of reactants have a great impact on the size distribution and dispersion of nano-ZnO. The diameter and dispersion of the products which were prepared with the optimal reaction conditions, which were obtained by the former phase's experiment, were improved. The diameter of nano-ZnO , which can be seen from the TEM image shown in Fig. 5, was between 20 and 40 nm.

3.3 The functional finishing of fabric

As shown in table 1, the impact of antistatic function finishing on the fabric whiteness is not obvious. The charge density of the treated fabric decreased significantly in comparison with the original piece, show that the fabric, which finished with the antistatic finishing agent was compounded with nano-ZnO, produce antistatic performance. And low concentration of finishing agent is able to achieve better antistatic effect. However, with the increasing of the addition amount of nano-ZnO, the fabric antistatic property is decreased, that is because of the declined dispersion and increased agglomeration of nano-ZnO in finishing agent. And the increased amount of nanoparticles results in the limitation of characteristics of nano-ZnO.

Through comparison between cotton and polyester treated fabrics, the decline rate of charge density of the later is more obvious than the former, it reveals that the antistatic effect of polyester fabric finished with nanometer antistatic finishing agent is better.

4. Conclusions

(1) The best reaction conditions for the preparation of nano-ZnO by direct precipitation method were as follows: The time of ultrasonic vibration was 40 min, the reaction temperature was 60 ℃, the concentration was 0.5 mol/l and the ratio was 1:2.

(2) The cotton and polyester fabrics treatment with nanometer antistatic finishing agent could significantly improve their antistatic properties, and low concentration of finishing agent could achieve better antistatic effect. The comparison between the two kinds of treated fabrics showed that the finishing effect of the polyester fabric was more obvious.

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Table 1. Effect of nanometer antistatic finishing agent on various fabrics hunter whiteness and charge density

Specimen	Cotton fabric		polyester fabric	
	Hunter whiteness	Charge density ($\times 10^7$) /(c·m^{-2})	Hunter whiteness	Charge density ($\times 10^7$) /(c·m^{-2})
0	93.71	36	84.56	58
1	89.63	5.7	85.35	0.95
2	90.13	6.9	86.48	7.1
3	90.40	8.4	87.45	14

0— original piece

1— the fabric was finished by 0.5% nano-ZnO, surfactant and binder

2— the fabric was finished by 1.5% nano-ZnO, surfactant and binder

3— the fabric was finished by 3% nano-ZnO, surfactant and binder

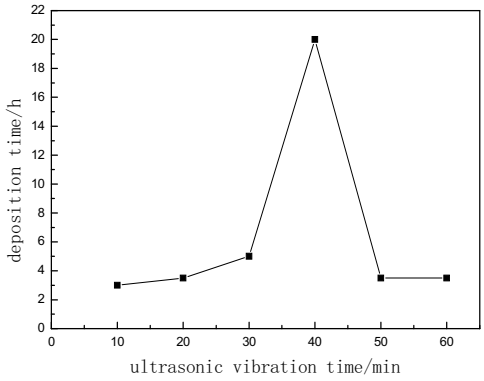

Figure 1. Effect of ultrasonic vibration time on product deposition time

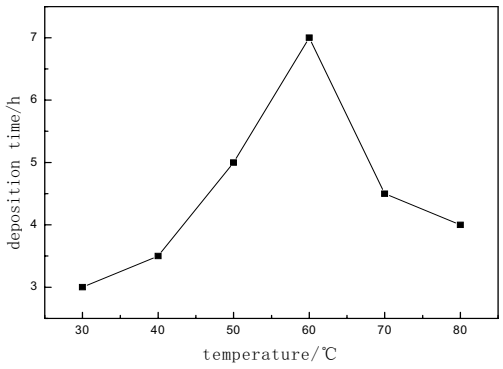

Figure 2. Effect of reaction temperature on product deposition time

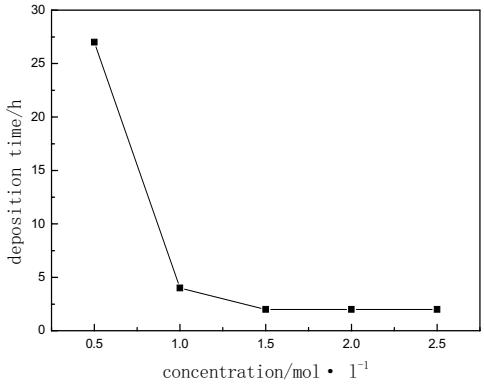

Figure 3. Effect of concentration of the zinc chloride on product deposition time

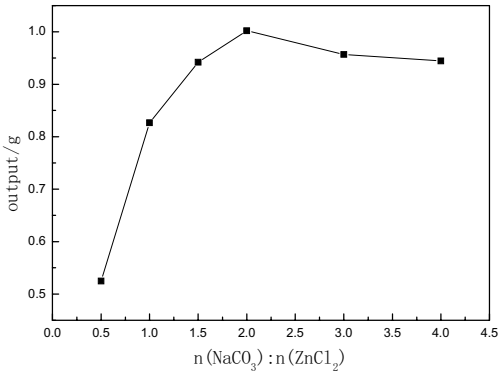

Figure 4. Effect of ratio of the reactant on precursor output

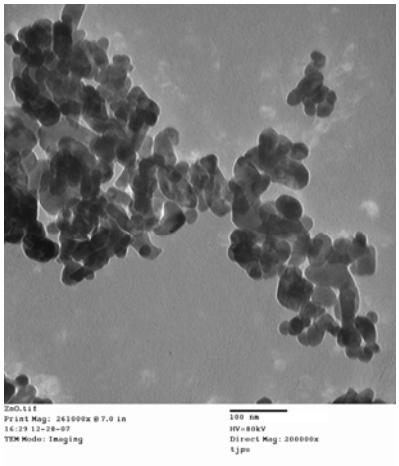

Figure 5. TEM photograph of nano-ZnO