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Effect of zinc oxide loaded polyester fibers on the color, mechanical properties and antibacterial action of woven fabric

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Abstract

The development of antimicrobial fabrics using zinc oxide (ZnO) embedded polyester fibers have been investigated in the present work. The mechanical properties, tear and tensile strength, of the fabrics have been investigated. The ZnO nanoparticles change the mechanical behavior of the fabrics. The antimicrobial activity of the fabrics was assessed through the bacterial reduction method and the materials showed activity against S. aureus. After three replicate standard washing processes, the bacterial inhibition of fabrics with about 99% reduction still remains. The fabrics exhibit great potential to be used as clothing textiles to protect human body against microbial infections.

1. Introduction

Nowadays, hygiene and well-being concerns have created a high demand for antimicrobial textiles to protect mankind from pathogens and to avoid cross infection. Examples for antimicrobial textiles in hygienically demanding areas are working clothes, socks [1], bedsheets and pillows [2], sportswear [3], hospital gowns [4-5], and even curtains [6]. The antimicrobial agents can be directly added to the spinning dope before extrusion [7] or applied to the textile substrate as a finish by exhaust [8-9], pad-dry-cure [5], coating spray [10], and foam techniques [11]. The chosen method is determined by a variety of factors including final use of the fabric, the capability of the manufacturer, and budget.

Antibacterial agents can be grouped into two types according to their chemical composition: organic and inorganic agents [12]. Most of organic antibacterial agents have limited their application because of poor heat resistance, high decomposability, and short life expectancy. Otherwise, inorganic antibacterial agents such as metal and metal oxides are advantageous compared to organic compound due to their stability and low toxicity [13-14]. As a result, inorganic antibacterial agents have received more recognition in their antibacterial product market [15]. Zinc oxide (ZnO), copper oxide (CuO), magnesium oxide (MgO), and titanium dioxide (TiO_2) are some of the most commonly used inorganic metals oxides. Among these metal oxides, ZnO has attracted a special attention as antibacterial agent.

ZnO, as one of the multifunctional inorganic nanoparticles, is listed as "generally recognized as safe" by the U.S. Food and Drug Administration (FDA) [12]. ZnO is widely used in sunscreens, coatings, and paints due to their UV absorption efficiency and transparency to visible light [16]. Besides, ZnO exhibits a wide range of antibacterial activities against both Gram-positive and Gram-negative bacteria [17] such as *E. coli* [18], *S. aureus* [19-20], *C. jejuni* [21], *S. typhimurium* [22], and *B.subtilis* [20]. However, very few detailed studies were conducted on the addition of ZnO into the spinning dope before extrusion to give permanent antibacterial effect.

The purpose of this study was to investigate the effect of different zinc oxide loaded polyester fiber compositions of the fabric. Color, tear strength, tensile strength, and antibacterial activity were used to evaluate the fabrics.

2. Experimental

2.1 Materials

Zinc oxide loaded polyester staple fiber, 38 mm in length, was donated from Perma Corporation, Thailand. The concentration of ZnO in fibers was fixed with 500 ppm. Cotton and polyester fibers were donated from Erawan Textile Co., Ltd, Thailand. All chemicals used were analytical grade and were used as received without any further purification.

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2.2 Fabric weaving and finishing

In this study, eight woven fabrics were produced by using SL8900 evergreen rapier machine from CCI Tech, Inc., Taiwan that the fabric composition are given in Table 1. Fabric construction was maintained for twill 2/1 fabric. These fabrics were purified by desizing and scouring with an aqueous solution containing 2 $g\cdot L^{-1}$ of α-amylase and 1 $g\cdot L^{-1}$ of wetting agent using a liquor ratio 1:30 at temperature 95ºC for 20 min. The fabrics were washed thoroughly with boiling water then with cold water. After that, all fabrics are bleached with an aqueous solution containing 3 $g\cdot L^{-1}$ of hydrogen peroxide (H_2O_2), 2 $g\cdot L^{-1}$ of sodium hydroxide (NaOH), 2 $g\cdot L^{-1}$ sodium carbonate (Na_2CO_3) using a liquor ratio 1:30 at temperature 95ºC for 45 min, the bleached fabrics were washed thoroughly with hot and then cold water and finally air dried. All fabrics were finished by padding fabric through 30 $g\cdot L^{-1}$of a solusoft® silicone softener from Archroma (Thailand) Company Limited, Thailand to a wet pickup of about 60%.

2.3 Color measurement

The whiteness and CIE L*a*b* values of fabrics were determined by using Datacolor Check II spectrophotometer, Datacolor, USA.

2.4 Scanning electron microscope (SEM) study

The morphologies of fibers and fabrics are examined by using a Jeol JSM-6400 scanning electron microscope. The samples were mounted on the SEM sample stub using a double-sided sticking tape. The samples were sputter coated with gold prior to SEM observation.

2.5 Mechanical properties

After preparation of test specimen, all fabrics are tested in the standard testing atmosphere 50 ± 2% relative humidity and temperature 23 ± 2ºC for 24 h prior to test. Tear strength was measured on conditioned fabrics using the Elmendorf tester according to the ASTM D 1424-09 test method. A template was used to cut fabric strips of 100 ± 2 mm length and 63 ± 0.15 mm width. The observation was recorded from the scale of the tester. The tear strength was then calculated by using the following formula:

$$\text{Tear strength} = 64 \times \text{scale reading}$$

Both warp and weft strips were tested for each sample and five observations were taken in each case.

Tensile strength was determined in a Testometric testing machine, model M350-5AT (Testometric Co., Ltd, England), according with ASTM D 5034 with a testing speed of 10 $mm\cdot min^{-1}$. Testing fabric has been prepared with a dimension of 300 mm × 25 mm. Each test has been performed in both warp and weft directions, after which the average values could be worked out.

2.6 Washing process

To evaluate the quality of antimicrobial fabrics, the permanence against mechanical and chemical stress was studied applying washing and drying cycles. The investigation was performed using an aqueous detergent solution as well as subsequent rinsing and drying steps in a modified wash test according to DIN EN ISO 105-C10, method A. The Linitest washing tester was used for determining the wash durability of the fabrics. The specimen with a size of 10 cm × 6 cm was treated with aqueous solution containing 5 $g\cdot L^{-1}$ of a detergent in a steel container at 40°C for 30 min using a liquor ratio 1:30. Then, the samples were transferred to a container with 2 L of distilled water and rinsed by careful moving. After that, the samples were rinsed under running cold water and dried in the air.

Table 1. Fabric composition.

Fabric sample number	Detail
1	100% cotton
2	90% cotton 10% ZnO loaded polyester fiber
3	80% cotton 20% ZnO loaded polyester fiber
4	70% cotton 30% ZnO loaded polyester fiber
5	55% cotton 45% ZnO loaded polyester fiber
6	100% ZnO loaded polyester fiber
7	55% cotton 45% polyester fiber
8	100% polyester

2.7 Antibacterial activity

The antimicrobial activities were measured against *S. aureus* by the determination of the size of the inhibition zone after 24 h of incubation at 37 ± 2°C. This test was performed according to the JIS L 1902: 2015 (Qualitative) technique.

Quantitative assessment of antibacterial activity exhibited on fabrics was carried out by AATCC Test 100-2004. Briefly, the fabrics were introduced in the 100 ml nutrient broth inoculated with the *S. aureus* microbe which is an opportunistic human pathogen causing community and hospital-associated infections [23] and incubated at 37°C for 24 h. Microbial inhibition was determined by the reduction in number of bacterial colonies formed with respect to the control sample using the following equations:

$$R = \frac{B - A}{B} \times 100 \qquad (1)$$

where
R = Percent reduction in bacteria
A = CFU for treated test specimen swatches in the jar incubated for 24 h contact period
B = CFU for untreated control test specimen swatches in the jar immediately after inoculation (at “0” contact time)

3. Results and discussion

3.1 Morphological studies

The SEM images of polyester and ZnO loaded polyester fibers were shown in Figure 1.

The SEM images show the characteristic structure of each fiber type (Figure 1). The polyester showed a smooth surface, while the ZnO incorporated polyester fibers showed increased surface roughness. This refers to the increased surface roughness due to the ZnO particles.

3.2 Color

One of the most important features of textile material is color. Color is the result of the physical modification of light by colorants as detected by the human eye and interpreted in the brain [24]. The CIE L*a*b* color scale is used to provide standard, approximately uniform color scale so that color values could be easily compared. The CIE L*a*b* and whiteness value of sample fabrics was shown in Table 2.

Three values of CIE L*a*b* are L* (white-black), a* (red-green), and b* (yellow-blue). The increased values of L*, a*, and b* define whiter, more reddish, and more yellowish, respectively.

The results in Table 2 show that all fabrics gave almost the same L* value, however, a* and b* values were not the same. The a* values increased while the b* values decreased with increase in the content of ZnO incorporated polyester fibers. The slight decrease in whiteness index of fabrics can be observed when the ZnO dosage applied is increased. These can be due to the dark brown to black color of ZnO masterbatch.

3.3 Tear strength

Tear strength is the resistance of the fabric against tearing or force required to propagate the tear once it is initiated. The tear strength is required in high performance applications as well as in the conventional textiles. The warp and weft directional tear test results on various fabrics are presented in Figure 2. Figure 2 shows the tear strength results of twill weave fabrics. It is seen that warp tear strength is significantly higher than weft direction due to higher number of warp yarns per inch than that of weft yarn.

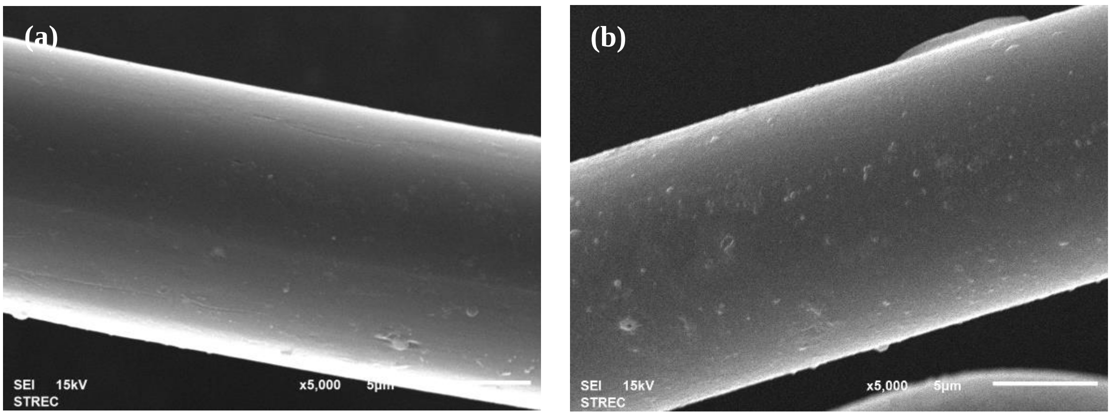

Figure 1. SEM images of (a) polyester and (b) ZnO loaded polyester fibers.

Table 2. CIE L*a*b* of various fabrics at different fiber type composition.

Fabric sample number	Whiteness index	L*	a*	b*
1	62.14	84.481	2.012	-7.137
2	114.98	84.350	2.381	-9.094
3	113.38	84.542	2.520	-9.692
4	111.00	83.871	3.066	-10.103
5	110.34	83.662	3.820	-10.339
6	106.78	83.498	3.837	-10.505
7	115.08	83.234	1.218	-5.435
8	144.47	83.477	1.045	-3.345

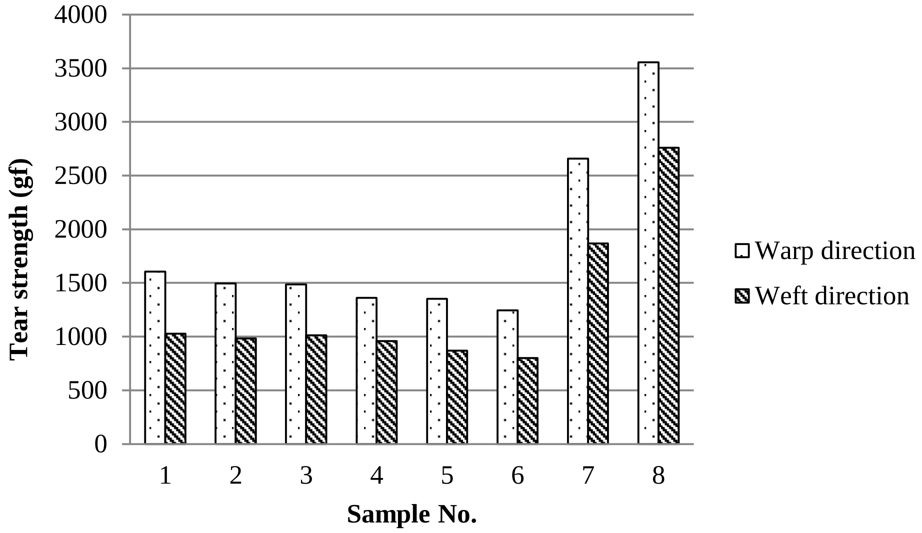

Figure 2. Tear strength of various fabrics at different fiber type composition.

It is observed that the fabrics made of ZnO loaded polyester fibers are less tear resistance. The neat polyester fabric experienced maximum tear strength of all. The overall results show that tear strength of ZnO loaded polyester/cotton fabrics was decreased by increasing ZnO loaded polyester content.

3.4 Tensile strength

Measurement of tensile properties is the most common mechanical measurement on fabrics. It is used to determine the behavior of a sample fabric while under an axial stretching load. From this, the breaking load can be obtained in Figure 3.

As seen in Figure 3, the neat polyester fabric presented the highest tensile strength in both warp and weft direction which are 660 and 457 N, respectively, while the neat cotton fabric showed the lowest tensile strength. This can be explained that polyester, a synthetic fiber has better tensile strength than cotton that is a natural fiber. Also, the tensile strength of warp yarn is higher than weft as warp yarns are stressed more during weaving. Moreover, the number of warp threads per inch is also higher than weft in woven fabric. As a result woven fabric shows higher strength in warp direction than weft.

The blending of ZnO loaded polyester component with cotton for the staple fiber spinning process (Sample No. 5) leads to decrease in fabric strength compared with polyester fibers combined with cotton (Sample No. 7). The phenomenon is associated with the decrease of polyester macromolecule arrangement along the fiber axis possibly caused by the antibacterial materials added in the structure [25]. It is seen that the tensile strength is not very much affected by amount of ZnO embedded in polyester fiber in the range studied. For higher ZnO loaded polyester fibers contents, there is a slight increase in tensile strength. These results imply that ZnO loaded polyester fibers loadings has little effect on tensile strength of fabrics and the reason may be caused by increased polyester content in fabric.

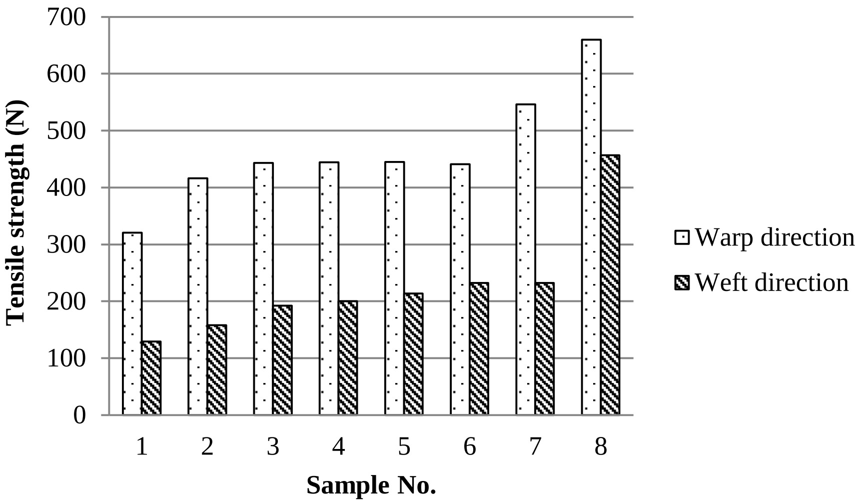

Figure 3. Tensile strength of various fabrics at different fiber type composition.

3.5 Antibacterial efficiency

The finished fabrics were evaluated for antibacterial activity against *S. aureus* before and after each laundering cycle up to three times according AATCC 100-2004 standard. The efficiency of antibacterial fabrics and the morphology of fabric before and after washing 3 times are shown in Figures 4-5, respectively. In addition, Figure 6 shows as example of antimicrobial activities against *S. aureus* by formation of a zone of inhibition around fabric samples.

SEM images of cotton-ZnO loaded polyester blend fabric samples before washing (Figure 4(a)) and after it has been washed at 40°C for 30 min with detergent solution for three times (Figure 4(b)). The formation of pills was observed on the fabric surface after the washing process, which affects the antimicrobial activity. It is obvious that the washed fabric has undergone silicone softener removals compared to unwashed fabric resulting in the enhancement of its antibacterial activities (Figure 5).

For Sample No. 2-4, the antibacterial properties of fabrics did not show any reduction with no wash. The 3rd wash results for reduction of *S. aureus* shown in Table 3 indicate that the increase amount of ZnO loaded polyester fibers in fabric, antibacterial property was progressively enhanced. It retained its antibacterial activity in the range of 83.13 to 99.86% even after 3rd launder cycle. There was 0% reduction in antibacterial efficiency without washing due to finished silicone softener on surface of fabrics. After 3rd laundering, the antibacterial activity is increased indicating that ZnO are well embedded inside the polymer matrix. It is interesting to note, that all fabrics showed no zone of inhibition (Figure 6). The results also showed that the antibacterial effect of fabrics occurred without migration of the active ingredient.

Between cotton and ZnO incorporated polyester fibers in the ratio 55: 45 (Sample No. 5) showed higher reduction rates than that of cotton/polyester fabric at the same ratio (Sample No. 7). It showed 30.15 and 0% reduction rates without any laundering and after three washing showed more than 99.94 and 0% reduction rates for Sample No. 5 and 7, respectively. This can be explained that ZnO incorporated polyester fiber content affects to antibacterial activity. Antibacterial ability of ZnO is a result from the attachment of ZnO to bacterial cell walls and subsequent release of Zn^{2+} ion to the bacterial cytoplasm [26].

4. Conclusions

Weaving from various compositions of cotton and ZnO loaded polyester fiber was prepared and studied. An increasing the amount of ZnO loaded polyester fibers caused changing color to more bluish and reddish. The tear strength of fabrics tended to decrease, while tensile strength of these fabrics was slightly increase with increasing ZnO loaded polyester fiber content. The antibacterial efficacy and washing stability of fabrics depends on the composition of the fiber type. Biocidal fabrics potentially will be effective in reducing, or eliminating entirely, pathogenic microorganisms and odor-causing micro-organisms which directly contact them.

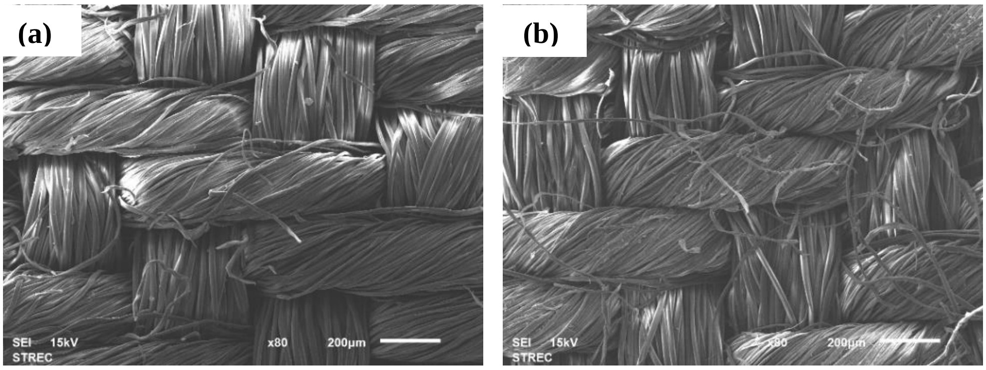

Figure 4. SEM images of the 70: 30 cotton-ZnO loaded polyester blend fabric samples (a) before and (b) after three times of washing.

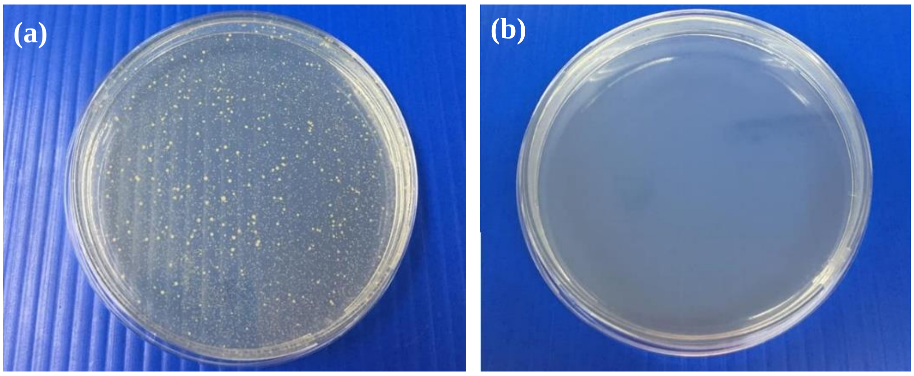

Figure 5. Images of the petri dishes showing antibacterial activity of the 70:30 cotton-ZnO loaded polyester fiber against *S. aureus* (a) before and (b) after three times of washing.

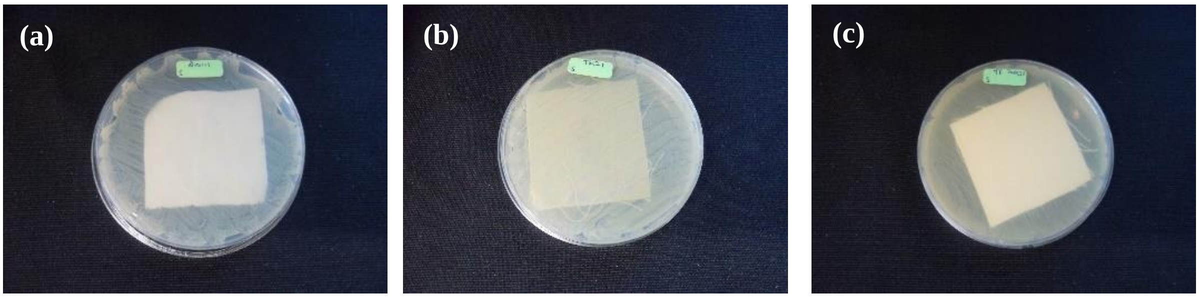

Figure 6. Images of samples tested according with the standard method JIS L 1902 (a) cotton fabric (b) 55:45 cotton-polyester blend fabric (c) 55:45 cotton-ZnO loaded polyester blend fabric.

Table 3. Antibacterial efficiency against *S. aureus.*

Fabric sample number	% reduction		Zone of inhibition (mm)
	No wash	3rd wash	3rd wash
1	0	0	0
2	0	83.13	0
3	0	99.63	0
4	0	99.86	0
5	30.15	>99.94	0
6	>99.99	>99.99	0
7	0	0	0
8	0	0	0

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