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# Nano ZnO or $TiO_2$ Provides Antibacterial Activities on Dyed Polyester Fabrics with Some of Azo Disperse Dyes

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**Abstract**
There is a growing interest in adding value to textiles through the use of zinc oxides or titanium dioxide at nano-particles. In the current study, the antimicrobial properties of six disperse dyes that we prepared previously against Gram-negative and Gram-positive bacteria and fungi were studied. The results show that zinc oxide or titanium dioxide in the nano size are able to reduce microbial growth in an excellent manner.
***Keywords***: *Disperse dyes, antimacrobial activities.*

## 1. Introduction

Azo dyes are the largest and most widely used industrial organic dyes used in a variety of applications such as textiles, paper, high-tech materials, leather, pharmaceuticals, foods, polymers and paints, [1-16]. Incorporating antimicrobial qualities into fabrics is becoming more popular. A number of studies have been published in which disperse dyes have been examined for antibacterial and antifungal activity with the goal of determining their potential medical use [5, 15].

A number of previous studies that we carried out described the use of metal oxides in nano size in treating polyester fabrics in order to increase the stability of these fabrics to light and give them the property of self-cleaning. [17-19], the current investigation focused on the use of zinc oxides or titanium dioxide in nano-size to treat polyester fabrics in order to give those fabrics an antimicrobial properties.

## 2. Materials and Methods

General method for the Synthesis of disperse dyes **1-6** which applied in this survey had been annotated in our published study [17-19].

***Dyeing procedure***

El-Mahalla El-Kobra Company, Egypt, provided scoured and bleached 100% polyester fabric. The disperse dyes **1-6**, a dispersion of the dyes were produced by dissolution of the appropriate amount of dyes (3% shades) in 2 ml DMF and then added drop wise with stirring to the dye bath (liquor ration 1:30) containing dispersing agent. The pH of the dye bath was adjusted to 5.5, and the wetted-out polyester fabrics were added. We performed dyeing by raising the dye bath temperature to 130°C at and holding it at this temperature for 60 min. After they were cooled to 50°C, the dyed fibers were rinsed with cold water and reduction-cleared (1 g/L sodium hydroxide, 1 g/L sodium hydrosulfite, 10 min, 80°C). The samples were rinsed with hot and cold water and, finally, air dried [17].

***Treatment of fabrics***

***Pre-treatment***

Fabric samples were soaked in a 10 g/l nonionic detergent solution (Hostapal, Clariant) for 10 minutes before being dispersed with $TiO_2$ NPs (0-2.5 percent w/w) for 15 minutes with gentle stirring. The materials were squeezed to eliminate excess dispersion before being dried in a 70°C oven for 10 minutes. The fabrics were queried for 3 minutes at 140 degrees Celsius. The fabrics were washed at 60 ºC for 15 minutes in an aqueous solution with a liquor ratio of 1:50 containing 3 g/l nonionic detergent solution (Hostapal, Clariant) [18].

***Post-treatment***

After dyeing, the fabric samples were soaked in a 10 g/l nonionic detergent solution (Hostapal, Clariant) for 10 minutes before being dispersed with $TiO_2$ nano particles (0-2.5 percent w/w) under gentle stirring for 15 minutes. The materials were squeezed to eliminate excess dispersion before being dried in a 70°C oven for 10 minutes. The fabrics were queried for 3 minutes

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at 140 degrees Celsius. The fabrics were washed at 60 $^{o}$C for 15 minutes in an aqueous solution with a liquor ratio of 1:50 containing 3 g/l nonionic detergent solution (Hostapal, Clariant) [19].

***Antimicrobial Activity Test***

The processes of evaluating the polyester fabric dyed with disperse dyes against four different pathogenic microbes was displayed according to the Agar well diffusion method that we have published before in our previous work [15].

**Determination of antibacterial activity by measuring colony forming unit (CFU).**

The antimicrobial activities of treated fabrics have been studied using colony forming technique (CFU) against *Staphylococcus aureus* and *Escherichia coli*. Bacterial stocks (100µl of stock of CFU value of about$10^8$) were inoculated into a 20ml freshly prepared liquid nutrient broth containing 5g/l peptone; 3g/L beef extract at pH 6.8 in 100 ml-volume of Erlenmeyer flasks, and incubated for 24h. Fabrics (250mg) were added to the inoculated flasks (with 20 µl of inoculums) leaving the control (inoculated flasks without samples).

After 24h incubation at 37$^{o}$C, the reduction (R%) in growth was measured by detecting the reduction in the absorbance of the inoculated flasks related to the untreated controls. The growth reduction was calculated from the following equation:

$$R (\%) = (A-B)/A \times 100$$

Where A is the absorbance of the culture control but B is the absorbance of treated sample

## 3. Results and Discussion

It is a great value to mention here that scheme 1 shows the chemical structure of the disperse dyes **1-6** used to dye the polyester fabrics [17-19].

### 3.1 Agar well diffusion technique

In our previous research, we presented important studies on the antimicrobial activity of azo disperse dyes [5-15]. The antimicrobial activity of the dyed polyester fabrics dyed the azo disperse dyes **1-6** was evaluated against Gram-positive (*Staphylococus aureus*) bacteria, Gram-negative (*Echerechia coli*) bacteria, and fungi (*Asperigilus fumigates* and *Candida albicans*) using the diffusion technique. The results revealed that all dyed fabrics do not show any growth inhibition zone against the tested microorganisms (*C.f.* Figures 1-4).

### 3.2 Colony forming unit (CFU).

An accurate quantitative evaluation of antimicrobial activity is possible on the fibre by colony counting. The antimicrobial activities of the treated fabrics have been studied. The results are shown in Tables 1 and 2. All the dyed fabrics with disperse dyes **1-6** displayed antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli*. In general, it is clear from Table 1, regarding the antimicrobial activities that treating polyester fabrics after the dyeing process (post-treatment) with nano-particles of zinc oxide Zn NPs was better than treating polyester fabrics before the dyeing process (pre-treatment), for all disperse dyes **1-6**, and the values of antimicrobial activities for untreated dyed fabrics were always lower than values of antimicrobial activities for treated dyed fabrics for all disperse dyes **1-6**.

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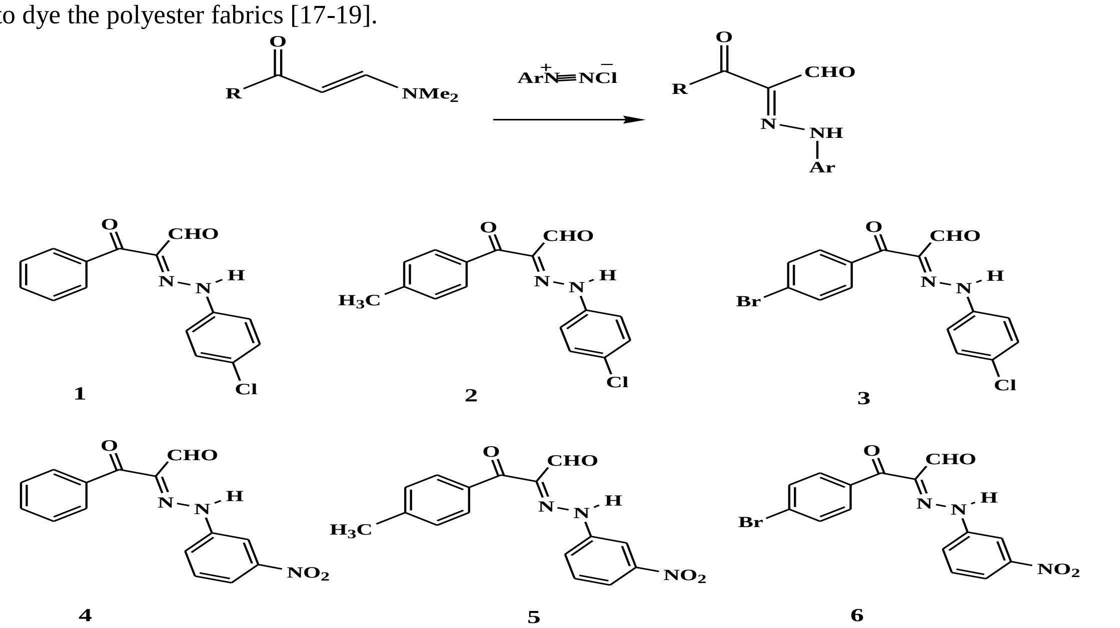


**Scheme 1**. Chemical structures of the disperse dyes 1-6

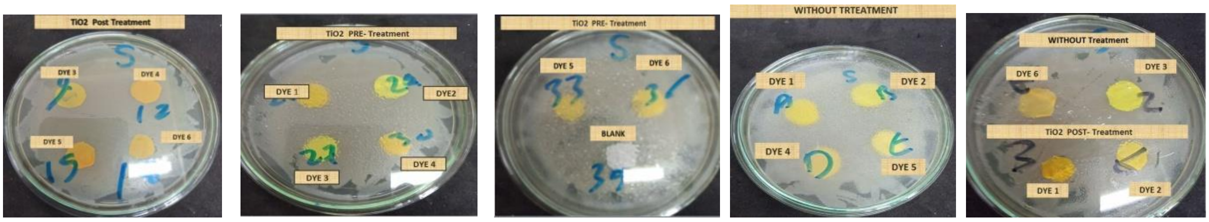
Figure 1. Staphylococus aureus micro organisms

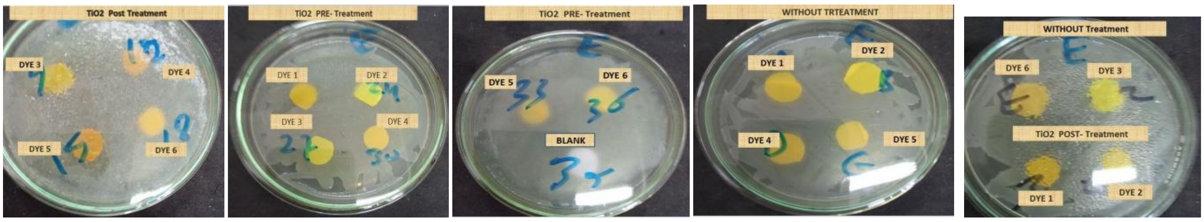
Figure 2. Echerechia coli micro organisms

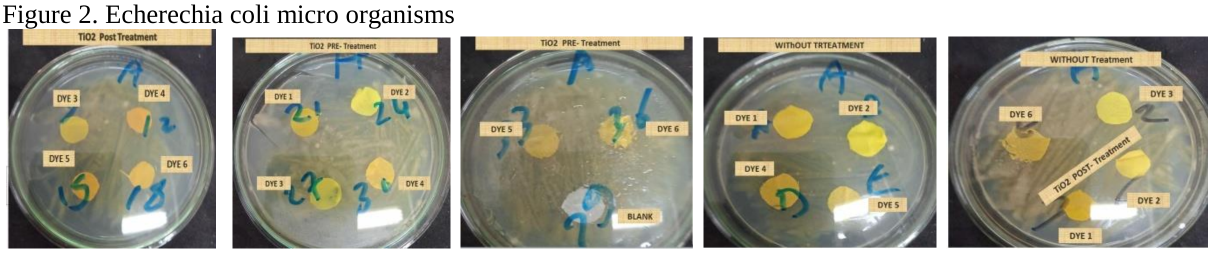
Figure 3. Asperigilus fumigatus micro organisms

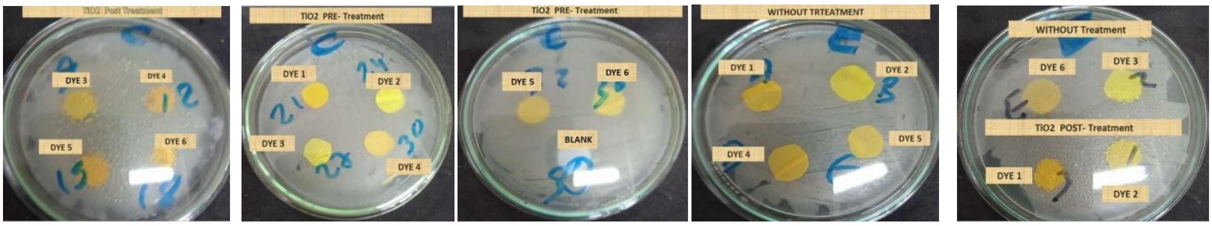
Figure 4. Candida albicans microorganisms

**Table 1.** Antimicrobial activities of the dyed polyester fabrics treated with ZnO NPs

| **Type of Treatment** | **Dyed Polyester fabrics with Dye No.** | **CFU by Absorbance method (A at 600nm)** | | | |
|---|---|---|---|---|---|
| | | ***Staphylococcus aureus*** | | ***Escherichia coli*** | |
| | | **Absorbance at 600nm** | **Reduction (R%)** | **Absorbance at 600nm** | **Reduction (R%)** |
| Without Treatment | Dye 1 | 0.048 | 97.75 | 2.037 | 00.00 |
| | Dye 2 | 0.836 | 60.79 | 1,864 | 05.00 |
| | Dye 3 | 0.229 | 89.26 | 1.926 | 00.00 |
| | Dye 4 | 0.298 | 86.02 | 1.854 | 05.00 |
| | Dye 5 | 0.043 | 97.98 | 1.827 | 07.00 |
| | Dye 6 | 0.031 | 98.55 | 1.830 | 07.00 |
| | Blank | 0.971 | 54.46 | 2.037 | 04.00 |
| Pre-treatment With ZnO NPs | Control | 2.002 | - | 1.964 | - |
| | Dye 1 | 0.063 | 96.95 | 2.002 | 00.00 |
| | Dye 2 | 0.037 | 98.21 | 1.889 | 3.82 |
| | Dye 3 | 0.044 | 97.87 | 1.874 | 4.58 |
| | Dye 4 | 0.095 | 95.40 | 1.832 | 6.72 |
| | Dye 5 | 0.098 | 95.25 | 1.882 | 4.18 |
| | Dye 6 | 0.040 | 98.06 | 1.897 | 3.41 |
| | Dye 1 | 0.028 | 98.64 | 1.832 | 6.72 |

| | | | | | |
|---|---|---|---|---|---|
| Post-treatment With ZnO NPs | Dye 2 | 0.024 | 98.84 | 1.588 | 19.14 |
| | Dye 3 | 0.025 | 98.79 | 1.855 | 5.55 |
| | Dye 4 | 0.019 | 99.08 | 1.791 | 8.81 |
| | Dye 5 | 0.043 | 97.92 | 1.813 | 7.69 |
| | Dye 6 | 0.010 | 99.52 | 1.804 | 8.15 |

**Table 2.** Antimicrobial activities of the dyed polyester fabrics treated with $TiO_2$ NPs

| **Type of Treatment** | **Dyed Polyester fabrics with Dye No.** | **CFU by Absorbance method (A at 600nm)** | | | |
|---|---|---|---|---|---|
| | | ***Staphylococcus aureus*** | | ***Escherichia coli*** | |
| | | **Absorbance at 600nm** | **Reduction (R%)** | **Absorbance at 600nm** | **Reduction (R%)** |
| Without Treatment | Dye 1 | 0.048 | 97.75 | 2.037 | 00.00 |
| | Dye 2 | 0.836 | 60.79 | 1,864 | 05.00 |
| | Dye 3 | 0.229 | 89.26 | 1.926 | 00.00 |
| | Dye 4 | 0.298 | 86.02 | 1.854 | 05.00 |
| | Dye 5 | 0.043 | 97.98 | 1.827 | 07.00 |
| | Dye 6 | 0.031 | 98.55 | 1.830 | 07.00 |
| | Blank | 0.971 | 54.46 | 2.037 | 04.00 |
| Pre-treatment With $TiO_2$ NPs | Control | 2.132 | - | 1.960 | - |
| | Dye 1 | 060 | 97.19 | 2.036 | 00.00 |
| | Dye 2 | 0.068 | 96.81 | 2.079 | 00.00 |
| | Dye 3 | 0.078 | 96.34 | 1.944 | 01.00 |
| | Dye 4 | 0.069 | 96.76 | 1.989 | 00.00 |
| | Dye 5 | 0.047 | 97.80 | 1.957 | 00.00 |
| | Dye 6 | 0.039 | 95.64 | 2.000 | 00.00 |
| Post treatment With $TiO_2$ NPs | Dye 1 | 0.023 | 98.89 | 1.881 | 4.23 |
| | Dye 2 | 0.015 | 99.27 | 1.934 | 1.53 |
| | Dye 3 | 0.035 | 98.30 | 1.897 | 3.41 |
| | Dye 4 | 0.040 | 98.06 | 1.878 | 4.38 |
| | Dye 5 | 0.043 | 97.92 | 1.844 | 6.11 |
| | Dye 6 | 0.018 | 99.13 | 1.874 | 9.00 |

Moreover, the data reveal that dyed polyester fabric with dye No. **2** has the most antimicrobial properties compared to the other disperse dyes **1**-**5**. Dyed polyester fabric with disperse dye No. **2** has the ability to show 99.52% reduction in CFU against *Staphylococcus aureus*.

In addition, treated polyester fabrics dyed with dyes **1**-**6** provided higher antibacterial efficacy against Gram-positive bacteria (*Staphylococcus aureus*) than Gram-negative bacteria (E*scherichia coli*), which could be attributed to the structural differences between two kinds of bacteria.

It is clear from Table 2, regarding the antimicrobial activities that treating polyester fabrics after the dyeing process (post-treatment) with nano-particles of titanium dioxide $TiO_2$ NPs was better than treating polyester fabrics before the dyeing process (pre-treatment), for all disperse dyes **1**-**6**, and the values of antimicrobial activities for untreated dyed fabrics were always lower than values of antimicrobial activities for treated dyed fabrics for all disperse dyes **1**-**6**.

Besides, the data reveal that dyed polyester fabric with dye No. **6** has the most antimicrobial properties compared to the other disperse dyes **1**-**5**. Dyed polyester fabric with disperse dye No. **6** has the ability to show 99.27% reduction in CFU against *Staphylococcus aureus*.

Additionally, treated polyester fabrics dyed with dyes **1**-**6** provided higher antibacterial efficacy against Gram-positive bacteria (*Staphylococcus aureus*) than Gram-negative bacteria (*Escherichia coli*), which could be attributed to the structural differences between two kinds of bacteria.

Hence, when comparing the antibacterial values of both techniques of treating dyed polyester fabrics with dyes **1**-**6**, we can state that ZnO NPs treatment is superior to $TiO_2$ NPs treatment

## 4. Conclusions

There is a growing interest in adding value to textiles through the use of zinc oxides or titanium dioxide at nano-scale. In the current study, the antimicrobial properties of six dispersed dyes that we prepared previously against *Staphylococcus aureus* as Gram-positive and *Escherichia coli* as Gram-negative bacteria were studied.. The results indicated that the treated sample with nano ZnO or nano $TiO_2$ have an excellent antibacterial activity against *Staphylococcus aureus* and a fair results against *Escherichia coli*.

## 5. Conflicts of interest

There are no conflicts to declare.

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