COLORING AND CHARACTERIZATION OF COTTON TEXTILES PLATED WITH SELF-CLEANING NANOPARTICLES.

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ABSTRACT
Cotton textiles tinting of by direct and reactive dyes using consumption method has been done by nanoparticles technicality. Gadolinium doped Zinc oxide nanoparticles (Gd\textsuperscript{3+}/ZnO) were successfully coated onto the surface of the cotton fabrics. ZnO and (Gd\textsuperscript{3+}/ZnO) nanoparticles are synthesized by precipitation method. The morphology, microstructure and functional groups were characterized for both of them through X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive analysis X-ray spectra (EDX), and FTIR spectroscopy. The diameter distribution of ZnO and (Gd\textsuperscript{3+}/ZnO) nanoparticles were confirmed by (TEM) analysis, the average size of particles was found to be 100 nm, 50 nm respectively. The dye exhaustion percentage (E%) and color strength (K/S) of direct orange 85 and reactive red 195 onto treated cotton fabrics is generally better than that obtained by using untreated samples under the same conditions. The overall fastness properties to rubbing, light, perspiration, washing for the colored samples are ranging from very good to excellent. Nano treatment improves the cotton surface and decreases the environmental impacts and fabric damage.

Key words: Cotton fabric, ZnO and (Gd\textsuperscript{3+}/ZnO) nanoparticle, Direct dye, Reactive dye.

1. INTRODUCTION
Reactive and direct dyes are anionic in character and water soluble due to the presence of sulphonic groups in the chemical structure. Since cotton fabric an anionic surface charge in water, these dyes have low intrinsic affinity for the fibre. The repulsive charge between dye and cotton can be overcome by adding an electrolyte, which has the effect of screening the surface charge on the fibre. The large quantity of salt causes pollution and does disturb the biological balance [1]. In the conventional dyeing, a large amount of energy is consumed, but some new techniques and methods for saving energy and time were investigated [2].

Nanotechnology is a fast emerging future’s technology, where the nano-sized materials have attracted increasing interest due to their unique structure and features [3]. Among many applications of nanotechnology that have environmental implications, owing to their outstanding properties, is the nanostructured semiconductors have comprehensive applications in the green chemistry areas such as wastewater treatment and self-cleaning surfaces [4].

Zinc oxide nanoparticles (ZnO NPs) is among the few nano-semiconductors non-toxic compound that have good chemical, mechanical, photostability and oxidation properties. This nanomaterial has been studied and widely used as a photocatalytic material for many environmental implementation such as wastewater and self-cleaning surfaces [5,6].

In this thesis, we aim to study the action of doping with lanthanide on physicochemical features of ZnO nanoparticles in different applications such as wastewater treatment and self-cleaning application [7].

The treatment of cotton fabric with inorganic nanoparticles before dyeing process proved to be more uniform, rapid, efficient, also increasing the dye uptake and improving the fastness properties of fabric towards light, rubbing, washing and perspiration. It is considered a better alternative to the conventional technique due to the reduction of environmental impact, and energy consumption [8].
2. EXPERIMENTAL TECHNIQUES :

2.1 Materials :

2.1.1 Chemicals :

All chemicals that used in the experimental works were at highly purified from Sigma-Aldrich (U.S.A), El- Naser (Egypt), LOBA For Chemical Company (India).

2.1.2 Cotton fabric :

Cotton fabric is supplied by Miser Company for spinning and weaving, Mehala El-Kubra, Egypt, was scoured in aqueous solution with a liquor ratio1:50 containing 2 g/l nonionic deterge solution and 2 g/l sodium carbonate (Na\textsubscript{2}CO\textsubscript{3}) at 50ºC for 30 min to remove impurities (wax and fat), then thoroughly rinsed in cold tap water, and dried at room temperature.

2.1.3 Dyestuff :

The following dyestuffs under investigation are direct and reactive, their names are given below:

2.1.3.1 Reactive Red 195 (RR 195)

Was kindly supplied by Merk7(Germany), the chemical structure of RR195 was shown below: (Mol.wt. = 1136.31 g/mol; \( \lambda_{\text{max}} = 542 \) nm)

2.1.3.2 Direct Orange 85 (DO85):

Was kindly supplied by Merk (Germany), the chemical structure of DO85 was shown below: (Mol.wt. = 1188.93 g/mol; \( \lambda_{\text{max}} = 483 \) nm)

2.2 Preparation of ZnO and 2%wt Gadolinium doped Zinc oxide nanoparticle (Gd\textsuperscript{3+}/ZnO) NPs:

1- To the synthesis typical of (Gd\textsuperscript{3+}/ZnO) NPs using precipitation method [9], in the 100 ml of bi distilled water 2 g of Zn(acetate)\textsubscript{2}.2H\textsubscript{2}O (99.9\%) was added and whiskered for 30 min. and then 5 ml of glacial acetic acid (99 \%) was added and further whiskered for 10 min.

2- Then 2wt \% of Gadolinium nitrate Gd(NO\textsubscript{3})\textsubscript{3}.6H\textsubscript{2}O under stirring for 10 min. was applied to the above mixture as dopant.

3-In the above-prepared solution, 15 ml of an optimized 4 M NaOH solution was added to change pH at 11.0 as was developed and stirred for 1 h with precipitate. at 160ºC in Teflon-lined stainless-steel autoclave.

4- The products were taken out then cooled at room temperature and washed with bdistilled-water until they were neutral to remove some existing ions and impurities.

5- The last products were dried at 80ºC.then the white products were calcined in muffle at 400ºC for 2 hours [10].Pure ZnO NPs was likewise prepared without doping for comparison.

2.3 Fabric Treatment with ZnO and Gadolinium doped Zinc oxide nanoparticle (Gd\textsuperscript{3+}/ZnO) NPs :

The fabric samples were soaked separately under gentle magnetic stirring for 30 min. in 2-propanol (98\%) dispersion of ZnO NPs and 2% wt Gadolinium plated Zinc Oxide nanoparticles. The fabrics were pressed to eliminate the excess dispersion and dried under
atmospheric pressure (dry heat) in an oven at 130°C for 15 min [11].

2.4 Dyeing procedures of untreated and treated cotton fabrics:

The treated and untreated fabric are dyed in a quos medium with an initial concentration 30 ppm of two dyes and liquor ratio (1:40) adding sodium sulphate Na₂SO₄ (30g/l) and sodium carbonate Na₂CO₃ (40g/l), dye solution put in water bath to adjust temperature. Dyeing process is carried out at (50, 70 and 90°C). At the end of dyeing, the dye bath is required to cool down and subsequent absorbance measurement. The absorbance measured at λ_{max} of each dye by JENWAY UV-Spectrophotometer to be ready for exhaustion percentage E% and colour strength (K/S) value calculation of exhaustion percentage E% according to the following equation [12]:

\[ E\% = \frac{A_0 - A}{A_0} \times 100 \]  

Where A₀ and A are absorbance of the dye solution in the dye bath originally and at time t at λ_{max}, respectively.

The reflectance values of each dyed fabric were measured at the λ_{max} and the corresponding colour strength (K/S) value were calculated using Kubel ka-Munk equation [13]:

\[ K/S = \frac{(1-R)^2}{2R} \]  

Where:

R = a decimal fraction of the reflection of the dyed fabric; K = the absorption coefficient; and S = the scattering coefficient.

The dyed samples has been removed, and rinsed in distilled water, so we can dry outdoors to be ready for the determination of cotton fabric properties.

2.5 Surface Characterization:

Field Emission Scanning Electron Microscope (FE-SEM), Energy-dispersive analysis X-ray analysis (EDX), Transmission Electron Microscope (TEM), Fourier Transform Infrared-Spectroscopy (FTIR) and Diffraction analysis (XRD) Measurements were used for surface characterization.

2.6. Determination of Cotton Fabric Properties:

The untreated and treated dyed cotton samples at dyeing recipe were tested for various fastness properties such as washing was studied according to the process ISO 105-C02 [11], rubbing was studied by way of the test process ISO 105-X12., perspiration according to test process ISO 105-E04 and light accordance with test process ISO 105-B02 [13].

3. RESULTS AND DISCUSSION:

3.1. Characterization of ZnO and Gadolinium Doped Zinc Oxide (Gd³⁺/ZnO) NPs:

3.1.1. X-ray Diffraction analysis (XRD):

Figure 3 a,b shows the XRD diffraction patterns of ZnO NPs (a) and (Gd³⁺/ZnO) NPs (b). For ZnO NPs Figure 3a, the peaks of diffraction in XRD showed the nanocrystalline shape and pure ZnO with hexagonal wurtzite shape [14,15]. For (Gd³⁺/ZnO) NPs, no peaks of diffraction of Gd³⁺ or additional impurities were found in Figure 3b, suggesting that Gd³⁺ ions will replace the Zn²⁺ sites uniformly in the ZnO lattices. These peaks at scattering angles (2θ) of 31.77, 34.40, 36.22, 47.50, 56.60, 62.63, 66.39, 67.95, 69.06, 72.56, and 76.97 which can be allocated to diffraction from: 100, 002, 101, 102, 110, 103, 200, 112, 201, 004, and 202 crystal planes, respectively as Figure 3. The higher intensities of calcined nanostructure peaks with smaller widths indicate a strongly crystalline structure of the wurtzite.

3.1.2 Scanning electron microscope (SEM) analysis:

SEM is one of the sample topography methods and provides important information on the mechanism of growth, constitute and size of the particles [16]. The morphology of the ZnO NPs and 2%wt (Gd³⁺/ZnO) NPs surfaces are shown in Figure 4 a,b.
The entire SEM picture shows clearly the mean size of the nanoparticles in order of nanometer scale. The ZnO NPs show in synthesized nanoparticles in Figure 4a is homogeneous, evenly spread over the surface and good contact between the spherical and randomly directed particles. In ZnO NPs and (Gd$^{3+}$/ZnO) NPs the tendency is noted for heavy agglomeration. Figure 4b shows the surface morphology of (Gd$^{3+}$/ZnO) NPs the samples which have slightly lesser particles size than the ZnO.

3.1.3 Energy-dispersive analysis X-ray analysis (EDX):

The energy dispersive analyses X-ray (EDX) is used to evaluate the quantity of components [16] in ZnO NPs and (Gd$^{3+}$/ZnO) NPs as shown in Figure 5 a,b. It is clearly shown the strong peaks in the spectrum related to (Zn) Figure 5a, (Gd) Figure 5b, have the weight percentages of 85.24% , 1.75% respectively for ZnO NPs and (Gd$^{3+}$/ZnO) NPs as shown in the Table 1.

| Samples           | Weight percentage of the elements (%) |
|-------------------|---------------------------------------|
| O                 | 14.76                                 |
| Zn                | 85.24                                 |
| Gd                | ----                                  |
| (Gd$^{3+}$/ZnO) NPs | 5.87                                 |
|                   | 92.38                                 |
|                   | 1.75                                  |

3.1.4. Transmission electron microscope analysis (TEM):

The diameter distribution of ZnO NPs and (Gd$^{3+}$/ZnO) NPs. were observed in TEM.

Figure 6 a,b. The average particles size are 100, 50 nm for ZnO NPs and (Gd$^{3+}$/ZnO) NPs.
3.1.5. FTIR Spectrum analysis:

For ZnO NPs and (Gd³⁺/ZnO) NPs the functional groups were studied using FTIR spectroscopy. The FTIR spectra shown in Figure 7a the broad absorption band at ~3437 cm⁻¹ corresponds to the O–H stretching vibration of water present in ZnO NPs and the other transmission band at 2925 cm⁻¹ is allocated to a residual organic component [14]. The band at ~1641 cm⁻¹ which appears at wave length just half of 3437 cm⁻¹ have been allocated to the first overtone of fundamental stretching mode of −OH. The transmission band at ~1534 cm⁻¹ and ~1458 cm⁻¹ in both cases, the carbonyl group of the carboxylate ions that may remain adsorbed on the ZnO NPs surface is responsible as shown in the Table 2.

As Figure 7b shows the FTIR spectra of (Gd³⁺/ZnO) NPs which are almost similar to that of the ZnO NPs. The bands which show up at low frequencies around 420 and 500 cm⁻¹ are allocated to the bending vibration of the characteristic peak for Zn-O bonds. Doping Gd³⁺ in these limits does not lead to appear further new peaks. This is possibly due to the homogeneous dispersion of Gd³⁺ particles on the zinc oxide surface without any clusters formation [17].

| Assignments                  | Wave number (cm⁻¹) | ZnO NPs | (Gd³⁺/ZnO) NPs |
|------------------------------|--------------------|---------|----------------|
| H-O-H stretching             | 3437.49            | 3429.78 |
| Residual organic component   | 2925.48            | 2923.56 |
| -O-H stretching vibration    | 1641.13            | 1633.41 |
| Zn-O bending vibration       | 429.08, 504.29     | 413.65, 494.65 |
3.2 Characterization of treated Cotton fabric (morphology analysis):

Figure 8a shows the SEM image of cotton sample untreated cotton fiber and Figure 8b treated cotton fiber with ZnO NPs. The morphological structure of cotton treated specimen revealed a harsh and more crumple fiber surface relative to the control fabric. The precipitation of the finishing agent on the fibers would damage the surface due to the agents’ slight acidity.

Figure 8c show the SEM of cotton fabric treated with ZnO and (Gd$^{3+}$/ZnO) NPs of irregular form linked to the cotton fabric were agglomerated during the process. However, higher magnification of SEM images did show the existence of the metal oxides as illustrated in Figure 8 c, d and e.

Figure 8b clearly show that the clustered ZnO NPs observed agglomerated particles with their diameters within 0.35-1.50 pm. Particular agglomeration was assumed to be mainly due to the surface attraction between small particles. On the other hand, Figure 8 c, d and e illustrates that the Gd$^{3+}$ particles were joined to the fiber surface in the form of small agglomerated particles, i.e. the particles were agglomerated together with the diameters 0.23-0.63pm

Figure 8 d and e depict the SEM images of the cotton specimen dyeing with RR 195 and DO85 respectively, shows parallel ridges and grooves with twisted convolution. Magnified image of this fiber surface indicates the distance between these ridges and grooves are in the micrometer scale. After the colored fabrics was obtained at various stages of coloring to support the suggested mechanism of interaction of dye fibre.

3.3. Dyeing of Cotton Fabric:

When cellulosic fabric is placed in water, the random areas swell to produce small pores. This small pores enable the dye molecules to diffuse into the structure of the fabric [18-20].

Cellulose fabrics have a small negative charge because of the presence of certain carboxylic acid groups from oxidation in the main hydroxylic sites [21]. At a higher pH, some of the hydroxyl groups on the hydroxyl methyl side chains may also be ionized which significantly increasing the negative charge [22].

Reactive dyes will react with cotton fabric on heating under neutral conditions as shown in Figure 9.

The molecular structures of reactive dyes is similar to that of acid and simple direct cotton dyes, the characteristics of a reactive dye are the chromophoric class, the water solubility sulphonate groups, the reactive group, and the bridging group that either directly binds the reactive group to the chromophore or to some other part of the dye molecule. Each of these structural features will impact the dyeing and fastness properties.

Specific cotton colors with direct dyes have inherent substantivity. Its aqueous solutions
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usually dyes cotton in the presence of an electrolyte like NaCl or Na₂SO₄. Direct coloring does not involve the use of a mordant and the dyeing process is very simple, as its name implies. Direct dyes have good light fastness, but low wet fastness and poor color build-up. Figure 10 shows Direct Orange 85 dye binds to cellulose fiber.

![SEM images of cotton (a) untreated (b) coated ZnO NPs (c) coated (Gd³⁺/ZnO) (d) coated (Gd³⁺/ZnO) with RR195 dye (e) coated (Gd³⁺/ZnO) with DO85 dye.]

**Fig. 8:** SEM images of cotton (a) untreated (b) coated ZnO NPs (c) coated (Gd³⁺/ZnO) (d) coated (Gd³⁺/ZnO) with RR195 dye (e) coated (Gd³⁺/ZnO) with DO85 dye.

**Fig. 9:** Chemical structures of reactive dye (RR195) with cellulose.

**Fig. 10:** Chemical structures of Direct dye (DO85) with cellulose.
3.3.1. Colorimetric and color strength:

Color strength (K/S) values of untreated cotton and treated cotton fabric with ZnO NPs and (Gd$^{3+}$/ZnO) on cotton fabrics for (RR 195) and (DO85) dyes are shown in Table 3.4. The result indicated that the presence of metal nanoparticles increases the dye affinity towards the cotton fabric. Gd doped ZnO in cotton thus act as a mordent. The negatively charged dye anions joined towards the cotton fiber. The better coupling of the dye and fiber improvement the color strength (Gd$^{3+}$/ZnO) have the highest K/S value of the treated cotton samples which attributed the positive impact as a co-catalyst, enhancing affinity of adsorption of dye. The higher K/S value assisted dyeing can be due to breakdown of dye aggregates by ultrasonic energy at fiber surface and the establishment of resulting balance in a shorter period of time [23].

Table 3: Colorimetric data of the dyed cotton fabric was pretreated using (RR195) dye at 50, 70 and 90 °C.

| Sample (cotton fabric) | Temp. °C | Shades of sample | L*  | a*  | b*  | c*  | h    | k/s  |
|------------------------|----------|------------------|-----|-----|-----|-----|------|------|
| Untreated              | 50       |                  | 72.82 | 21.63 | -8.68 | 23.31 | 338.14 | 8.60 |
| ZnO                    | 50       |                  | 77.12 | 17.65 | -6.39 | 18.77 | 340.10 | 11.39 |
| (Gd$^{3+}$/ZnO)        | 50       |                  | 76.51 | 18.12 | -5.96 | 19.07 | 341.80 | 14.41 |
| Untreated              | 70       |                  | 76.38 | 17.25 | -6.85 | 18.56 | 338.34 | 41.87 |
| ZnO                    | 70       |                  | 77.95 | 16.30 | -5.79 | 17.30 | 340.44 | 42.20 |
| (Gd$^{3+}$/ZnO)        | 70       |                  | 77.32 | 18.18 | -8.86 | 19.43 | 339.34 | 44.77 |
| Untreated              | 90       |                  | 72.55 | 23.16 | -9.64 | 25.09 | 337.41 | 63.71 |
| ZnO                    | 90       |                  | 80.19 | 13.45 | -4.95 | 14.33 | 339.81 | 78.94 |
| (Gd$^{3+}$/ZnO)        | 90       |                  | 81.03 | 13.42 | -4.53 | 14.16 | 341.34 | 80.71 |
3.3.2 Dye exhaustion %:

**Figure 11, 12** are showing the effect of dyeing temperatures for reactive and direct dyeing. The dyeing is conducted at different temperatures increased from 50 to 90 °C in presence of best dyeing conditions with untreated and treated cotton fabric ZnO, (Gd³⁺/ZnO) NPs with initial dye concentration (30ppm) and L.R 1:40 for one hour dyeing time for C.I. RR195 and C.I. DO85.

**Figure 11** shown the results of (E%) for (RR195) at maximum temperatures 90°C in untreated and treated cotton fabric ZnO, (Gd³⁺/ZnO) NPs respectively are (31.111, 48.231 and 89.714%).

**Figure 12** shown the results of (E%) for (DO85) at maximum temperatures 90°C in untreated and treated cotton fabric ZnO, (Gd³⁺/ZnO) NPs respectively are (58.061, 78.658, and 93.011%).

The reason for this trend is that beginning with the rise of temperature, molecular
structure of cellulose opens and thus dyeability is enhanced. Further, the increases of the temperature reduce the value of the substantivity ratio and increase the reactivity, both these factors reduce the dye adsorption. The decrease in the dye exhaustion at higher temperature is the hydrolysis of the dye prefer to react with the hydroxyl group of water [24].

3.3.3 Determination of cotton fabric properties

Color fastness properties of the untreated and treated ZnO, (Gd\textsuperscript{3+}/ZnO) NPs on cotton fabrics for (RR 195) and (DO85) dyes were measured as seen in Table 5,6. The results indicate good fair fastness properties of the treated with ZnO and (Gd\textsuperscript{3+}/ZnO) NPs dyed samples for the rubbing and washing while are very good for the untreated sample. There are no difference in perspiration and light fastness for the dyed fabrics either untreated or treated NPs which gives very good results [25].

As shown in Table 5, 6, the fastness to washing, rubbing and perspiration of all samples colored with the dye were to very good to good irrespective to the fabric used.

Fig. 11: Effect of dyeing temperature on (E%) for (RR195) at maximum temperatures 90\degree C values of dyed cotton fabric for 1 h.

Fig. 12: Effect of dyeing temperature on (E%) for (DO85) at maximum temperatures 90\degree C values of dyed cotton fabric for 1 h.
Table 5: Fastness properties of dyed cotton fabrics using (RR195) dye at 50°C.

| Cotton sample | Fastness to rubbing | Washing fastness | Fastness to perspiration | Light |
|---------------|---------------------|------------------|--------------------------|-------|
|               | Wet | Dry | Sw | Sc | Alt | Sw | Sc | Alt | Sw | Sc | Alt |
| Untreated     | 4-5 | 4-5 | 4-5 | 4-5 | 3   | 4-5 | 4-5 | 3   | 4-5 | 4-5 | 4   |
| (ZnO)         | 4-5 | 4   | 4-5 | 4   | 3   | 4-5 | 4-5 | 2-3 | 4-5 | 4-5 | 4   |
| (Gd³⁺/ZnO)    | 4-5 | 3-4 | 4-5 | 3-4 | 3   | 4-5 | 4-5 | 2-3 | 4-5 | 4-5 | 4   |

Table 6: Fastness properties of dyed cotton fabrics using (DO85) dye at 50°C.

| Cotton sample | Fastness to rubbing | Washing fastness | Fastness to perspiration | Light |
|---------------|---------------------|------------------|--------------------------|-------|
|               | Wet | Dry | Sw | Sc | Alt | Sw | Sc | Alt | Sw | Sc | Alt |
| Untreated     | 4-5 | 4-5 | 2  | 4-5 | 3   | 2  | 3  | 3   | 1  | 4  | 4   |
| (ZnO)         | 4-5 | 4-5 | 3  | 4-5 | 3   | 2  | 2  | 3   | 3  | 3  | 3   |
| (Gd³⁺/ZnO)    | 4-5 | 4-5 | 3  | 4  | 3   | 2  | 3  | 3   | 2  | 3  | 3   |

CONCLUSION:

1. ZnO and (Gd³⁺/ZnO) NPs were prepared by precipitation method and described by electron microscopy (SEM, TEM), energy-dispersive analysis X-ray spectra (EDX), and X-ray diffraction analysis.

2. ZnO and (Gd³⁺/ZnO) NPs can be suitable applied on cotton to produce shades having good color strength k/s and exhaustion percentages (E%).

3. ZnO and (Gd³⁺/ZnO) NPs treatment produces a cotton fabric with advanced color fastness enabling them to decrease the environmental impacts and fabric damage.

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تم صباغة الأنسجة القطنية باستخدام نوعان من الصبغة وهو الصبغة البرتقالية المباشرة 85 والصبغة الحمراء النشطة 195 في استخدام حبيبات نانوية. تم استخدام الحبيبات النانوية الجادولينيوم على أكسيز الزنك فأثبتت نجاحها في تعطي سطح الأنسجة القطن. وتم تحضير الحبيبات النانوية لكل من أكسيز الزنك والجادولينيوم على أكسيز الزنك باستخدام طرق الترسب. ودراسة استخدام التركيب الميكروكسيمي والمجموعات الدالة لصفات كلاً منها وذلك عن طريق استخدام اجهزة مختلفة وهي حيود الاشعة السينية (XRD) والميكروكسيموبوندروفي الماسح (SEM) والتحليل (EDX) الاستقصائي والمسببة في النيكلية والتحليل بواسطة كاميرات (FTIR). وقد تم التأكد من التوزيع النظري لكلا من أكسيز الزنك والجادولينيوم على أكسيز الزنك بواسطة جهاز (TEM). حيث وجد أن متوسط حجم الحبيبات النانوية 100-50 نانومتر لكل من المادات على الترتيب. وأظهرت الدورات أن نسبة استنفاد الصبغة وشدة اللون للصبغة البرتقالية المباشرة 85 والصبغة الحمراء النشطة 195 الناتجة على الأنسجة النانوية المعلقة أفضل بشكل عام من التي تم الحصول عليها باستخدام عبوات الأكسيز غير معلق تحت نفس ظروف. ومن تعين خصائص الحبيبات النانوية للحلك بالضوء والغسل والعلاجات المضخمة أثبتت تحسين من جيد جدا إلى ممتاز. وكذلك أثبتت الدراسات النانوية أنها حصدت أنسجة القطن وعملت على تقليل التأثير البيئي وتفت الأنسجة.