

Eco –friendly Flame Retardant Via Self Assembly Coating

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LIMITING oxygen index (LOI) of the most of the textiles in use is lower than 21%, while cotton has 18.4%, which means that they can burn easily once the fire has started. Currently, there is some difficulty in making durable flame retardant (DFR) treatments for cotton fabric using an ordinary one step wet process, which is nontoxic to humans and the environment. This research has also aimed to investigate the optimum treatment conditions that allow controlled deposition of zinc oxide onto carboxymethylated cotton fabric using sodium hypophosphite (SHP) /citric acid (CA). In this regard, the influence of the process parameters on the physicochemical and performance properties of treated cotton fabric will elucidate. The treated fabric was monitored for carboxylic content, LOI, char length, char residue, whiteness index, retained tensile strength and elongation at break. The formation of zinc oxide was verified using UV-Visible spectrum at wavelength of 325nm. Also, the zinc oxide in nano scale was monitored and observed in the range of 19-41nm by transition electron microscope (TEM). The treated cotton fabrics show LOI value and char length 25.3%, 3cm , respectively, compared with 18.4 and 11cm for untreated fabric. The existence of zinc oxide particle interaction and other functional groups introduced into cotton fabrics were promoted by Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM) and X-ray diffraction (XRD).

Keywords: Carboxymethylation, Cotton fabrics, Functional finishing, Flame retardant, LOI.

Introduction

In the last years, nanotechnology has attracted a great interest from both industrial and academic research, because of the encouraging and astonishing results achieved in numerous fields by employing nano-sized objects. There are different methods to enhance final properties of fibers and fabrics in particular, smart textiles displaying antimicrobial or UV radiation protection [1], dye fastness, wrinkle resistance finishing, super-hydrophobicity and photocatalytic properties [2-5]. At the present time, researchers focus on protecting the textile fabrics from ignition by using zinc oxide, titanium dioxide, phosphoric acid [6-8], behind surface modification after-treatments capable of changing or conferring different properties to the investigated textiles. The curing step has to be applied without any effect on the properties of the textile. For that, the surface has to be modified by formation of micro-to nano-sized coatings and synthesis novel coatings for fabrics and fibers. These coatings described by Layer by

Layer (LbL) which exhibit hybrid organic and inorganic composition or complete inorganic, can be made by using different approaches [9]. LbL was first described in 1966 [10-14]. The LbL is an encouraging technique by which an adsorption technique of nano-particles is created [10]. Simply, it is recognized as step-by-step film build-up based on electrostatic connections for polyanion/polycation couples to form polyelectrolyte multilayers [15]. This will be a good environment for the inorganic nanoparticles to demonstrate different connections (such as; hydrogen bonds, covalent bonds, etc.) next to the electrostatic one. Application of the LbL concept for fabric treatment of an electrostatic connection requires the alternate immersion of the fabric into an oppositely charged polyelectrolyte usually water-based solution or dispersion. As a result, the occurrence of positively and negatively charged layers piled up on the substrate surface, exploiting a complete surface charge reversal after every immersion step [16]. Recently, many articles, including the main role of LbL in the protection of fibers and fabrics from

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the ignition and the aim of synthesis a thermal insulator system [9,17]. Such barrier may operate as a thermal shield for the surrounding substrate, favoring such phenomena as char formation and inhibiting the production of volatile species that can increase the combustion (as; cellulose). Certainly, a thermal insulator barrier can support the dehydration of cellulose followed with char formation and inhibit its depolymerization toward the production of volatile species as furan and levoglucosan [18].

Materials and Methods

Materials

Gray Loomstate cotton fabric (100 %, cover factor=12.5), plain weave 1/1, 3/1 (Warp-way: 3/1 warp way twill) of weight 265 g/m² was supplied by the Misr Company for Spinning and Weaving, El Mehala El-Kobra, Egypt. Monochloroacetic acid (MCAA, C₂H₃ClO₂, 99%), citric acid (99%), and sodium hypophosphite were supplied from Sigma-Aldrich Company. Sodium hydroxide, sodium carbonate, sodium silicate, hydrogen peroxide (30%), orthophosphoric acid (85%), Egyptol® (non-ionic wetting agent based on ethylene oxide condensate), and methanol were supplied by El Gomhoria Company, Cairo, Egypt.

Desizing and scouring

The cotton fabric was desized and scoured by using an aqueous solution containing sodium hydroxide (6 %), Egyptol (2 g/l), liquor to goods ratio of 50:1 at 95°C for 30 min. After scouring, the specimens were washed thoroughly with water then dried at ambient temperature.

Bleaching

Scoured cotton fabric was treated with an aqueous solution of hydrogen peroxide (6 g/L), sodium silicate (2 g/L) and organic stabilizer (1 g/L), at pH 10.5 using aqueous sodium hydroxide. The liquor to goods ratio was 50:1 and the bleaching process was carried out at 95°C for 45 min. The fabric was washed with water at 100°C for several times (5 times) followed by washing with running cold water before drying at ambient temperature.

Preparation of nano zinc oxide

Nano zinc oxide was synthesized by sol-gel technique [19,20]. Unless otherwise stated before, the method was performed as follows: 0.01 mole of zinc acetate dihydrate was dissolved in 50 ml

methanol and heated at 50°C along with stirring for 1hr, thus making the precursor solution A. Solution B was prepared by dissolving 0.02 mole sodium hydroxide in 50 ml of methanol then heated at 50 °C with continuous stirring for 1hr. The ZnO nano-sol was synthesized firstly, by adding solution B into solution A with a dropwise, constant stirring at 50°C for 30 min, then the mixture kept stirring for 2 hr and cooled at room temperature. Subsequently, a homogenous and transparent sol was obtained. Finally, the precipitate was dried in an oven (TARKO 200) at 85°C for 4 hr and thermally treated at 450 °C for 3 hr to form nano zinc oxide.

Treatment of carboxymethylated cotton nano zinc oxide

Carboxymethylated cotton fabric (CMC) having a carboxyl content of 335meq/100 gm [21] was padded in an aqueous solution containing different concentrations of nano zinc oxide (3-6%), citric acid (3-7%) and sodium hypophosphite (4-7%). The treated fabric was dried at 85°C for 5min and cured at different temperatures (150-180°C) for varied time intervals (1-7min).

Testing and analysis

Determination of limiting oxygen index (LOI)

The determination of the lowest oxygen percentage need to ignite continues was carried out using an LOI instrument (Rheometric Scientific LTD, UK) according to standard test method, ISO 4589 [22]. The specimen (5 specimens) of cotton fabrics with dimension 5x15cm² was handled in U holder in a vertical position inside a thermal glass chamber. Both of oxygen (ignition gas) and nitrogen (as purification for oxygen gas from mixing with other gasses and lose its characteristics) gasses were used to ignite the specimen. Flame source was propane gas.

Char length

The char length was measured according to BS 3119 standard method [23].

Char residue

The weight loss of each specimen has been measured [24] by dividing W₂ by W₁ according to the following equation :

$$\text{Char yield (\%)} = \frac{W_2}{W_1} \times 100 \quad (1)$$

where W₁ and W₂ are the weight of specimens

before and after burning, respectively.

Tensile strength and elongation at break

The mechanical properties of the uncoated and coated specimens were tested on the Shimadzu Universal Tester of (CRT)- type S-500, Japan, according to ASTM D5035 [25]. Each specimen was cut in a warp direction with dimension 3.5x15cm². The average of the five specimens results is considered.

Whiteness index

The whiteness of the specimens before and after coating was measured by the UltraScan@ Pro Hunter lab spectrophotometer according to standard test method [26].

FTIR analysis

FTIR spectra were recorded using Nicolet 380 Spectrometer – USA, depending on the absorption of electromagnetic radiation in the frequency range 4000 to 400cm⁻¹, with an average of 32 scans by using a resolution of 4 cm⁻¹. A pressure of 18Kpa was derived to the crystal holder to make reproducible contact between the crystal face and the fabric. [27,28].

Transition electron microscope (TEM)

The Tecnai F12 TEM (Philips Electron Optics, Holland) was used to measure the zinc oxide nanoparticle size. Grid with size 97μm was used to prepare the test specimen by dropping 2 to 3 drops of zinc oxide nanoparticles on a 200 mesh formvar coated copper (grid size: 97μm) (Ted Pella, Inc., Redding, CA, USA), then the excess solution was removed before drying for 12hr to image particles.

Scanning electron microscope (SEM)

JEOL JSM-840A (Tokyo, Japan) scanning microscope at an accelerating voltage of 15 kV was used to study the surface morphology of cotton fabrics and zinc oxide nanoparticles loaded cotton fabrics.

UV-Visible spectrophotometry

The UV-visible spectra of ZnO suspended in deionized water were recorded in Specord 50 ANALYTIKJENA® spectrophotometer, from 200–800nm, then measured after sonication.

XRD analysis

X-ray diffraction was used to analysis the specimens by using Phillips®PW 1710 X-Ray

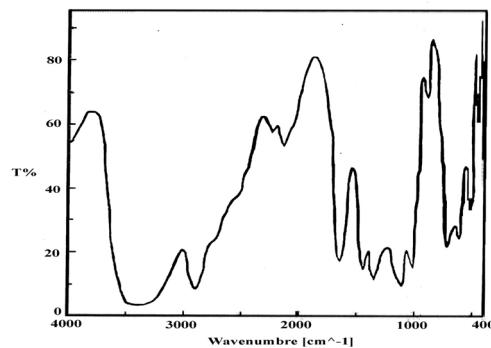
Diffractometer. The instrument including nickel-filtered CuKα ($\lambda = 1.54 \text{ \AA}$). The diffracted intensities and radiation were recorded from 30–80° 2θ angles [29].

Results and Discussion

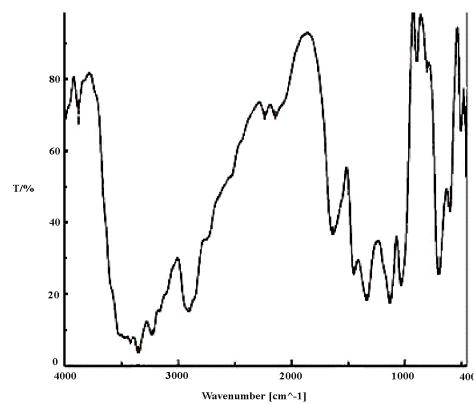
Characterization of the prepared zinc oxide nano particles (ZnO NPs)

Fourier transform infrared

The FTIR spectrum of carboxymethylated treated fabrics is shown in Fig. 1, compound A. In addition to the cellulose peaks at new peaks indicate the presence of the following: carboxylic group: 3531cm⁻¹ (carboxylic O-H stretching); 1653cm⁻¹ (-C=O stretching of carboxylic acid); 1120cm⁻¹ (-C-O-C- asymmetric bridge stretching), 701 cm⁻¹ (-O-C=O bending in carboxylic acid) [21]. The FTIR spectrum of carboxymethylated specimens treated with nano zinc oxide as shown in Fig.1 compound B shows different absorbance peaks: 3880 cm⁻¹ (O-H free); 3349cm⁻¹ (O-H);



Compound A



Compound B

Fig. 1. FTIR compound A: is CMC and compound B: is CMC treated with nano zinc oxide

2906 cm⁻¹ (C-H); 1634 cm⁻¹ (C=C); 1450 cm⁻¹ (C-O); 503 cm⁻¹ is characteristic for Zn-O bond [30].

Transition electron microscope

Figure 2 (a-c) shows TEM images of nano zinc

oxide prepared by the sol-gel method. They show that zinc oxide particle size is having an average range between 19-41nm. The Figure illustrates that, the size of nano zinc oxide increased when added ZnO by 3%, while decreasing the

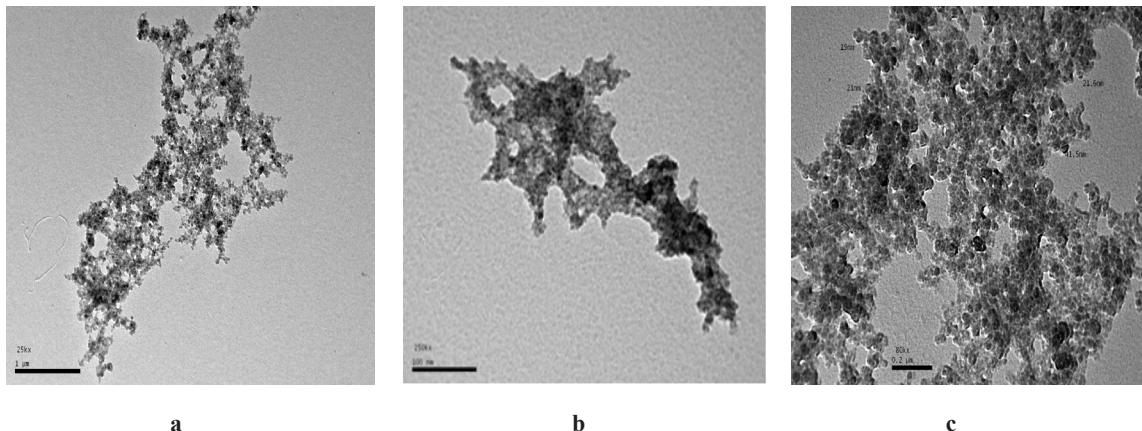


Fig. 2 (a-c). TEM image of nano zinc oxide particle with different particle size a; 19nm, b; 30nm, and c; with 40.5nm

concentration of the ZnO lead to incomplete growth of the ZnO nanoparticles.

Scanning electron microscope

The image as shown in Fig. 3 reflects the surface morphologies of carboxymethylated treated cotton fabrics with no observable difference. The images as shown in Fig. 3 (a-d) reflect the surface morphologies of

carboxymethylated fabric treated with nano zinc oxide. Indeed, morphological imaging confirmed that the treated fabric was predominately covered by a layer. The Figure illustrates that the surface of the untreated specimen is comparably rough, but the treated specimen surface appears much smoother, in order to nano zinc oxide coating which leads to the flattening fiber surface. The SEM image confirmed that the size of nano zinc

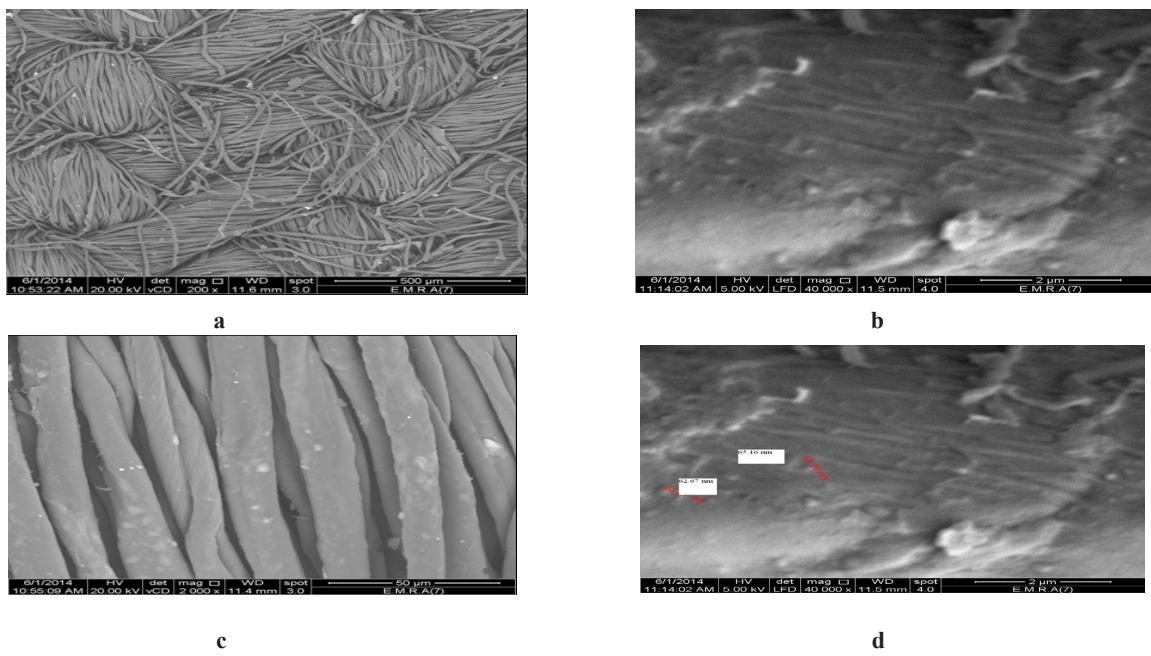


Fig. 3. SEM image of surface morphology of untreated cotton fabric (a-c), and carboxymethylated cotton fabric (d)

oxide particles existed in the surface was in the range of 62-65nm.

UV-Visible

Most of organic solvent (as; toluene) and water did not dissolve the zinc oxide powder (white crystalline). Hence UV-Visible spectra were recorded for the zinc oxide dispersed in methanol solution and also is represented in Fig. 4. The absorption band observed at 325nm is the characteristic peak of zinc oxide nano

material. The absorption peak is shifted to shorter wavelength compared to maximum absorption of zinc oxide occurs at 373nm. This shift is appropriate to the decrease in the size effect of nano structures. The approximate band gap value calculated from the λ_{max} value using the equation below [19].

$$\text{Energy band gap} = 1.2/\lambda_{\text{max}} \text{ (eV)} \quad (2)$$

The band gap value is calculated from the above equation which is equal to 3.6ev which is characteristic for nano zinc oxide.

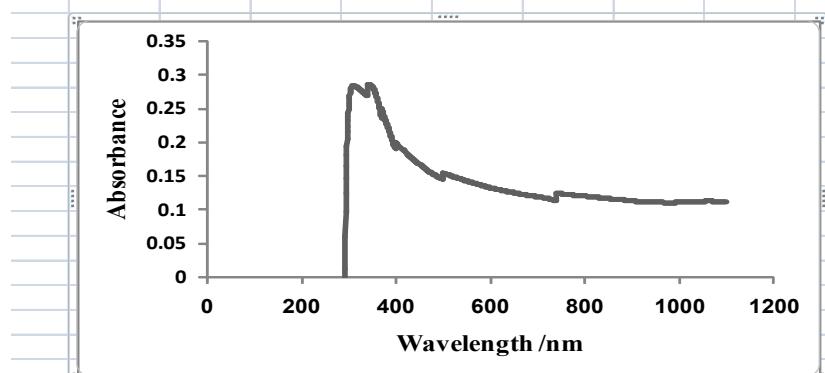


Fig. 4. UV-Visible curve of nano zinc oxide.

X-ray diffraction

Figure 5 depicts the X-ray diffraction (XRD) pattern of ZnO thin film deposited on the carboxymethylated treated cotton fabric. From the XRD pattern, one can clearly observe a diffraction peak at $2\theta=34.78^\circ$. The strong preferential increase is observed along C-axis, i.e. (002) plane indicating that the prepared ZnO nanocrystals have quartzite structure [31]. The unit cell "a" and "c" of the polycrystalline ZnO film with (002) orientation are calculated using the relation (3) and (4).

$$c = \lambda/\sin \Theta \quad (3)$$

$$c = \lambda/\sin \theta \quad (4)$$

The values obtained for the unit cell $a = 2.59850\text{\AA}$ and $c = 4.50$ are in good agreement with those reported in the JCPDS standard data (Card no. 80-0074). Where λ , and θ are the X-ray wavelength ($=1.5406\text{\AA}$), and Bragg angle respectively. Also, the XRD diffraction pattern shows three diffraction peaks at $2\theta = 15, 16.5$ and 22.9 .

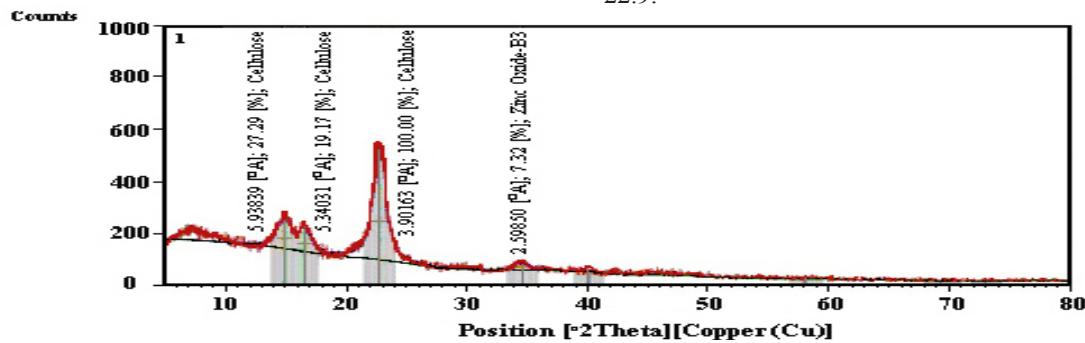


Fig. 5. XRD of CMC fabric treated with nano zinc oxide

Flame retardancy mechanism for treating fabric-tentative mechanism

In our paper, negative charge was located on the treated fabric as the carboxylate anion (COO^-) by carboxymethylation of cotton fabric and subsequently extended to a layer of nano zinc oxide. Using citric acid, which works at SHP and cross-linker, which act as a catalyst to raise the percent of nano zinc oxide bonded to the cured fabrics as shown in Fig. 6 [32].

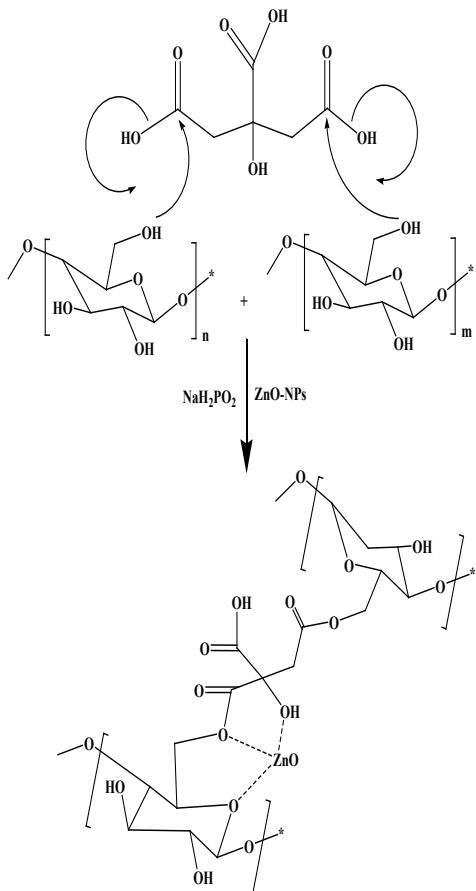


Fig. 6. Scheme mechanism between CMC with citric acid and nano zinc oxide

Effect of citric acid concentration

Table 1 illustrates that as the concentration of citric acid increased up to 4%, the limiting oxygen index increased up to 23.7% and char residue up to 64.07% while, char length decrease to 3cm. By adding citric acid with the highest concentration 7% lead to decrease the percentage of oxygen to the lowest value 22.3% less than 4% concentration with the char length up to 4.5%. These may be discussed that with further increasing the citric

acid to 4% all active sites occupied, so no reaction between citric acid and nano zinc oxide occur and by washing the physically adsorbed nano zinc oxide and citric acid removed and decreased all the flammability properties.

Increasing the acid concentration (up to 7%), decreases the tensile strength of the treated specimen to 50 and 18.0% for elongation. Finally, the whiteness recorded the highest value at cons. 4%, then start to decrease as cons., increase recording the lowest value due to the fabric deteriorate.

Effect of sodium hypophosphite

SHP is a phosphorus-based salt working as a catalyst for cross-linking cellulose with polycarboxylic acids [33,34]. It has a direct effect on improving the performance properties of the treated fabric and catalyzing ester-crosslinking reactions, which consume the carboxyl groups and buffering the cross-linking system. The fabrics treated with SHP is due to increase bonding to the adjacent carbons in their molecular backbones and both are able to esterify cellulose by first forming a reactive 5 membered cyclic anhydride intermediate [35] as shown in Fig.1.

Table 2 shows that increase in the concentration of sodium hypophosphite lead to improving the flammability properties of the treated fabrics. As the concentration of SHP increased to 7%, the LOI value increased to 25.5 and char residue increased gradually to 65.7%, whereas char length decreased to 3.0cm. These may be discussed that increasing the concentration of SHP increases more bonding formed between nano zinc oxide, citric acid and carboxylic group on the treated fabrics were more nano zinc complex formed on the treated fabrics. The mechanical properties improved by inter chemical reaction, since the elongation decrease (22.0%) the tensile strength increase (78.0Kg.f).

Effect of nano zinc oxide concentration

Table 3 illustrates the improvement in the ignition properties of the fabric, since adding nano zinc oxide with 3% concentration recording the highest oxygen index value (25.8%), ash residue value (69.2%) compared to the untreated specimen. ZnO nanoparticles have a direct effect of increasing the mechanical properties and whiteness index value (79.0%), due to the interchemical reaction achieved between cotton fabric and nano particles of ZnO .

TABLE 1. Effect of citric acid concentration of the properties of fabrics coated by nano zinc oxide layer. Conditions used: Carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 6% SHP, dried at 85°C for 5min, curing at 160°C for 3min. The fabrics were evaluated after five washings

Concentration citric acid /(%)	LOI /(%)	Char length /cm)	Char residue /(%)	Tensile strength /Kg.f)	Elongation at break /(%))	Whiteness index /(%))
Untreated	18.4	11.0	26.2	74.0	27.0	69.4
3	22.8	3.8	62.8	80.0	22.0	71.0
4	25.2	3.0	64.1	83.0	21.2	71.0
5	22.8	3.4	63.3	75.0	20.0	66.0
6	22.5	3.8	62.4	61.0	20.0	58.0
7	22.3	4.5	58.2	50.0	18.0	54.0

TABLE 2. Effect of sodium hypo phosphate concentration on the properties of fabrics coated by nano zinc oxide layer. Conditions used: Carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 4% citric acid, dried at 85°C for 5min, curing at 160°C for 3min. The fabrics were evaluated after five washings

Concentration SHP /(%))	LOI / (%))	Char length / (cm))	Char residue / (%))	Tensile strength / (Kg.f))	Elongation at break / (%))	Whiteness index / (%))
Untreated	18.4	11.0	26.2	74.0	27.0	69.4
4	21.9	4.7	61.3	79.0	22.0	71.0
5	22.6	3.6	63.2	79.0	22.0	71.0
6	25.2	3.0	64.1	83.0	21.2	71.0
7	23.5	3.0	65.7	81.0	22.0	71.0

TABLE 3. Effect of zinc oxide concentration on the flame retardancy and mechanical properties of fabrics. Conditions used: Carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 6% SHP, 4% citric acid, dried at 85°C for 5min, curing at 160°C for 3min. The fabrics were evaluated after five washings

Concentration of Zinc oxide /(%))	LOI / (%))	Char length / (cm))	Char residue / (%))	Tensile strength / (Kg.f))	Elongation at break / (%))	Whiteness index / (%))
Untreated	18.4	11.0	26.2	74.0	27.0	69.4
3	25.2	3.0	64.1	83.0	21.2	71.0
4	23.2	3.0	61.3	82.6	22.0	73.0
5	22.9	3.1	58.9	82.0	23.0	75.3
6	22.3	3.3	58.6	80.0	23.5	74.0

Effect of curing temperature

Table 4 shows that increasing the curing temperature from 150 to 160°C increase the oxygen index value up to 25.2% and char residue increase gradually up to 64.1% with char length decreasing 73% compared to the untreated

specimen. Increasing the curing temperature lead to form more nano zinc oxide and more complex of nano zinc oxide with citric acid to act as a thermal barrier layer to the treated fabrics. With further increase the curing temperature up to 180°C has a bad effect on the flammability properties as the

limiting oxygen index value decreased to 22.0% and char residue also decreased to 50.4%. These may discuss in that at higher curing temperature there were unsaturated acids formed which decreases the bonding between citric acid and carboxymethylated treated fabrics. Moreover,

Table 4 shows that as the curing temperature increases up to 180°C, the tensile strength decreased to 61.0 and whiteness index also decreased to 51.0 may due to discoloration and formation of unsaturated acid.

TABLE 4. Effect of curing temperature on the flame retardancy and physical properties of treated cotton fabrics.
Conditions used: Carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 6% SHP, 4% citric acid, dried at 85°C for 5min, cured for 3min at 160°C. The fabrics were evaluated after five washings

Curing temperature /°C	LOI / (%)	Char length / (cm)	Char residue / (%)	Tensile strength / (Kg.f)	Elongation at break /(%)	Whiteness index /(%)
Untreated	18.4	11.0	26.2	74.0	27.0	69.4
150	22.0	4.9	58.0	79.0	25.0	71.0
160	25.2	3.0	64.1	83.0	21.2	71.0
170	23.0	5.0	58.3	72.0	20.0	62.0
180	22.0	5.4	50.4	61.0	18.0	51.0

Effect of curing time

The curing time has a direct effect on increasing the value of LOI. Table 5 shows that increasing curing time from 1 up to 3 min, the value of oxygen index increased to 19.3% and 23.7%, respectively, the char residue up to 64.1% from the weight test, while the char length decreased to 2.8cm (at curing time 3 min). With further increased, the curing time up to 7 min the LOI has a slight decrease and

reached 23.3%, while char length 7.6 cm. These may be explained that at curing time 3 min, the more increase in the formation of nano zinc oxide, which imparts thermal barrier layer to the treated fabrics. Increasing the curing time has a negative effect on the tensile strength, elongation and whiteness index.

TABLE 5. Effect of curing time on the flame retardancy and physical properties of treated cotton fabrics.
Conditions used: Carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 6% SHP, 4% citric acid, dried at 85°C for 5min, cured for 3min at 160°C. The fabrics were evaluated after five washings

Curing time / min	LOI / (%)	Char length / (cm)	Char residue / (%)	Tensile strength / (Kg.f)	Elongation at break /(%)	Whiteness index /(%)
Untreated	18.4	11.0	26.2	74.0	27.0	69.4
1	22.8	5.7	63.9	82.0	23.0	71.0
3	25.2	3.0	64.1	83.0	21.2	71.0
5	23.8	4.8	64.2	65.0	17.0	57.0
7	23.3	7.6	64.3	55.0	16.0	51.0

Conclusions

Applying layer by layer (LbL) assembly technique for improving the flame retardant properties of cotton fabric without serious damage in the mechanical properties is our achievement in this work. The carboxymethylated cotton

fabric having carboxyl content 335 meq/100gm cellulose was treated with nano-ZnO (3-6%) and different treatment condition of curing time and temperature as well as, citric acid and SHP get an adequate balance between greatest flame retardant properties (represented as a highest LOI value) and least deterioration of fabric mechanical

properties (expressed as; tensile strength and elongation percentage). Optimum flame retardant properties (25.3% LOI) with lowest mechanical deterioration were obtained with the following conditions carboxylic content 335 meq/100gm cellulose, 3% nano zinc oxide, 6% SHP, 4% citric acid, dried at 85oC for 5min, cured for 3min at 160oC. The fabrics were evaluated after five washings. The obtained data were proved using SEM and XRD.

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مثبت لهب صديق للبيئة عن طريق التجميع الذاتي للطلاء

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١. الهدف من البحث:

المعروف أن القطن يدخل دوره رئيسي في صناعة المنسوجات القطنية بشكل عام وفي المفروشات وصناعة الملابس الخارجية والداخلية لإمتيازه بالراحة والنعومة وسرعة امتصاص العرق إلا إنه سريع الإشتعال ويتوجه مصاحباً بأدخنة كثيفة. لذا هدف هذا البحث إلى حماية المنسوجات القطنية من الإشتعال وذلك بتغطيته بالطبقة فوق الطبقة (LbL) بطلاء جديد دائم صديق للبيئة يعمل على تقليل سرعة إنتشار اللهب.

كما تناول هذا البحث إلى اختيار أفضل ظروف المعالجة من حيث تركيز حمض الستريك والصوديوم هيبوفوسفات وأكسيد الزنك ودرجة الحرارة الازمة لتجفيف العينات وكذلك زمن المعالجة.

٢. أهم الاختبارات:

- قياس المحتوى الكربوكسيلى لعينات القطن المختبرة.
- التحقق من تواجد أكسيد الزنك الثنوى المتكون على سطح العينات باستخدام الأشعة فوق البنفسجية.
- قياس الخصائص الميكانيكية لعينات بقياس قوة الشد والإستطالة٪.
- قياس معامل البياض والإصفرار.
- قياس الخصائص الإشتوعالية لعينات باستخدام جهاز قياس أقل نسبة أكسجين لازمة للإشتعال (LOI).
- تحديد الطول المتقدم من العينات المختبرة.
- قياس نسبة الوزن المتقدم الناتج من العينات.

٣. أهم النتائج:

أكذ تحلل العينات باستخدام FTIR وجود المجموعات الحديثة التي تم تغطيته سطح العينات القطنية المعالجة بها.

أكذ فحص العينات عن طريق TEM وجود جزيئات ZnO في مستوى النانو بأحجام جزيئات تتراوح بين 19-41nm.

أثبت المسح الإلكتروني الميكروسكوبى (SEM) أن سطح العينات المعالجة أكثر استواء ونعومة وذلك يؤكد وجود ZnO في صورة جزيئات نانوية عن مثيلاتها الغير معالجة والتي ظهر سطحها خشن وغير مستوى.

إنخفاض في درجة البياض بدون تغير مؤثر في الخصائص الميكانيكية لعينات المختبرة.

تحسين مقاومة العينات المعالجة للإشتعال لذلك ارتفع نسبة LOI من ٤٪١٨ إلى ٣٪٢٥.

إنخفاض الجزء المتقدم من 3cm إلى 11cm.

تم التوصل لأفضل ظروف المعالجة لعينات المقصول على أفضل مقاومة للإشتعال وكانت كالتالى: 335meq/100gm كربوكسى ميثيل سيلیوز (CMC) و ٣٪ أكسيد الزنك النانو و ٦٪ صوديوم هيبوفوسفات و ٤٪ حمض الستريك وتم التجفيف عند درجة حرارة 160°C خلال ٣ دقائق.