Egypt. J. Chem. **Vol. 62**, No. 6. pp. 1047 - 1055 (2019)

Egyptian Journal of Chemistry
http://ejchem.journals.ekb.eg/

Antimicrobial Polypropylene Loaded by Cubic Cuprous Oxide Micro Particles

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RECENTLY, nanotechnology of metals and their oxides is increased and became important for production of antimicrobial textiles. In situ synthesis of micro cuprous oxide particles (Cu_2O), 1-30 mM/L onto non-woven polypropylene and study the antimicrobial activity were investigated. Cu_2O micro particles were prepared by chemical method using copper sulphate pentahydrate, sodium hydroxide and glucose as a reducing agent at 90^o C for 2 hrs, followed by pad-dry-cure at 150ºC for 5min. Cu_2O content onto PP was estimated before and after five washings and drying. Characterization of Cu_2O Micro particles onto PP was done by XRD, SEM, EDX and which had revealed the presence of Cu_2O micro particles in cubic shape, with face dimeter 0.6-0.8μm. Characterization of polypropylene color before and after Cu_2O treatment was determined at λ_{max} 410 nm using the colorimetric data (L*, a*, b*, ΔE) and the color strength (K/S). The antimicrobial activity of treated PP against pathogenic microbes such *S. aureus, E. coli*and *C. albicans* was evaluated after 24 hrs contact time and gave very good antimicrobial results.

Keywords: Polypropylene (PP), Copper sulphate pentahydrate, Micro-cuprous oxide, Alkaline glucose, Antimicrobial activity.

Introduction

Nowadays antimicrobial textiles are very important and have a wide range of application for human demands due to the appearance of dangerous diseases in the world.

The present research work aims for the synthesis and loading of Cu_2O micro particles as an antimicrobial agent onto polypropylene (PP).

Literature review on the above subject revealed that new properties for antibacterial textiles were developed using nano particles which involved antibacterial activity, UV protection, increased fabric dyeability, flame retardancy, soil release, tensile strength and wrinkle resistance. In situ, eco-friendly synthesis of silver nano particles onto synthetic fabrics [polyester (PET), polyamide-6 (PA-6) and polypropylene (PP)] was carried out by reduction of silver nitrate to silver nano particles. SEM images had indicated the well distribution of silver nano particles onto PET and PP fabrics surface. The treated fabrics had outstanding antibacterial and antifungal properties against *Staphylococcus aureus*, *Klebsiella pneumonia* and *Candida albicans*. Furthermore, PA-6/nano Ag composite fabric had displayed electrical conductivity better than the untreated one [1].

Nano zinc oxide, titanium dioxide and aluminum oxide were used for finishing and adding new multifunctional properties to synthetic fabrics by pad-dry-cure method and they acquired good washing property. PP and PET fabrics were activated by dielectric-barrier discharge (DBD) plasma and then they were dispersed in a colloidal solution of isopropanol with nano particles of ZnO, TiO_2 and Al_2O_3(2or4%on weight fabric(owf))). PET fabric treated with nano ZnO had excellent antibacterial

*Corresponding author email: smgawish@yahoo.com
Received 20/5/2019; Accepted 13/6/2019
DOI: 10.21608/ejchem.2019.13001.1813

activity against *S.aureus* and *K.pneumoniae*. DBD plasma treated fabric had improved the binding of nano particles to the surface of PET and PP fabric due to increase the active groups on fabric surface. Activated PP fabric had better UV protection compared to the blank [2]. Copper and copper oxide have special properties, which are used in various technological applications such as superconductors, photoconductive applications, etc. Nowadays, they have been used as antimicrobial agents against a wide range of microorganisms [3-7]. Also, literature review revealed that nano copper was synthesized by the chemical reaction between copper salt and sodium hydroxide at100ºC. It was deposited onto polyester fabric and glucose was used as a stabilizer [8]. Antimicrobial active PP was achieved by adding copper metal and copper oxide nanoparticles into PP polymer matrix. Nano copper oxide was more effective as antimicrobial agent than Cu nano-metal [9]. Copper nanoparticles were synthesized and were applied onto cotton woven fabric by a pad-dry-cure method. The coated fabrics acquired antibacterial activity against *S.aureus* and with stand up to 25 wash cycles [10].

Polyester, cotton and polyester-cotton blend fabrics were treated with copper nanoparticles by a pad-dry-cure technique. The nano copper treated woven fabric was dyed with a natural dye and was found to improve both K/S and color properties [11]. The antibacterial coating durability of cotton and polymeric fabrics was increased by surface initiated grafting polymer which was introduced onto the substrate surface to bridge copper nanoparticles coatings and fabric. The fabric coated by copper nanoparticles had showed efficient antibacterial activity against *S.aureus* and *E.coli* even after 30 washing cycles. The copper nanoparticles were prepared onto the fabric by strong chemical bonds and with excellent durability [12].

Viscose fabric loaded with uniform micro-needles of Cu_2O was obtained by in situ reduction of copper salt. Fabric containing cuprous oxide had excellent antimicrobial activity against different microorganisms [13]. Under water diaphragm plasma was used to immobilize copper crystals onto PP surface and provide antibacterial activity [14]. PP/Copper NPs composites were prepared by the melt blender and they showed antibacterial activity [15, 16]. Pure copper nanoparticles were produced in the presence of chitosan as stabilizer by a chemical method. The antibacterial and antifungal activities of the nanoparticles were measured against several microorganisms. The size of the copper nanoparticles (2-250nm) were obtained depending on the concentration of chitosan stabilizer [17]. A sonochemical method was used for synthesis of cupric oxide (CuO) nanoparticles. Colloidal chitosan solution was prepared and mixed with CuO nanoparticles. The coated cotton fabric had showed enhanced antibacterial activity [18]. A simple and low cost method for in situ synthesis of copper nanoparticles onto cotton fabric was prepared by using a chemical reduction method.

Copper sulfate as a precursor, citric acid as a stabilizing and capping agent and sodium hypophosphite as a reducing agent were used. The excellent antibacterial activity of fabric containing copper nanoparticles after 30 washing cycles had confirmed their use in textile and medical products and had high stability for the treated fabric [19]. CuO nanoparticles were synthesized by precipitation method of different precursors such as copper nitrate $Cu(NO_3)_2$ and copper chloride $CuCl_2$. The formation of CuO nanostructures with different forms was prepared using different precursors by this method [20-22].

Stable polyvinyl pyrrolidone (PVP) with copper nanoparticles was prepared using glucose. They were found to be stable for two months. The stable and monodispersed copper nanoparticles were used in different applications [23-25].

Cu-containing fibers and fabric were prepared using copper–D-gluconate (Cu–DGL) complexes and sodium borohydride as reducing agent. Cu–DGL treated fabric had good antibacterial activity, even when low amounts of copper were present onto the fibers after washing [26].

In the following investigation, different concentrations at 1-30 m.M/L of $CuSO_4.5H_2O$ were reduced to Cu_2O micro particles by NaOH and glucose as reducing agent at 90ºC for 2 hrs, padded, dried and cured at 150ºC for 5 min. Characterization of PP fabric containing micro Cu_2O was done by XRD, SEM and EDX. Also, antimicrobial activity for PP treated fabric was evaluated.

Experimental

Materials

Fabric

Nonwoven polypropylene (20 g/m^2, thickness 2.60 mm) was a gift from Egyptex Co., Cairo.

Chemicals

Chemicals used were copper sulphate pentahydrate ($CuSO_4.5H_2O$), sodium hydroxide (NaOH), D-glucose ($C_6H_{12}O_6$), which were purchased from local market.

Methods

Scouring of fabric

Nonwoven PP fabric was well extracted with acetone before treatment to remove antioxidants and lubricants from the fabric.

Formation and deposition of cuprous oxide micro particles onto PP

A known weight of $CuSO_4.5H_2O$ (x gm) was dissolved in 160 ml distilled water, sodium hydroxide (0.8 gm/40ml distilled water) was added dropwise to 2 gm PP fabric in a stoppered conical flask, followed by addition of 1 gm glucose as a reducing agent (Table1). The solution was highly mixed in a shaking water bath. Then, the temperature was raised from 60ºC to 90ºC and maintained for two hours at that temperature. The fabric was padded (Roaches Co, England), dried at 100ºC for 30 min., cured at 150ºC for 5 min, weighed, washed five times and dried. The weight increase due deposition of cuprous oxide was calculated.

Analysis methods

Percent add - on cuprous oxide

Percentage weight cuprous oxide add-on the treated fabrics was calculated as follows (Eq.1):

$$\% \text{ wt } Cu_2O = [(W_2 - W_1) / W_1] \times 100 \quad \text{Eq.1}$$

Where, w_1: initial weight of the fabric; w_2: weight of the fabric after curing and washing

Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDX)

The fabric was well washed in distilled water to remove the unattached particles of micro Cu_2O and then dried before SEM determination. The fabric was measured on SEM (Tescan Vega 3 SBU) working at 20 KV. The fabric was coated with carbon double face and fixed with stubs of Quanta holder and examined in low vacuum.

Color measurements

Three coordinates (L*,a*and b*)of CIE LAB color system for color measurement of PP fabric at λ_{max} 410 nm were determined using Ultrascan Pro Hunter Lab. L* indicates the lightness of white- black axis and a* and b* show the redness–greenness and yellowness–blueness values, respectively. The color difference between two samples is determined by ΔE using Eq. 2 [27].

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5} \quad \text{Eq. 2}$$

ΔE: the total color difference between the untreated and treated fabric.

Fourier Transform Infrared Spectroscopy (FTIR)

Infrared Spectra were recorded using FTIR Spectrometer (Jasco FT/IR 4700). The transmittance between 400 and 4000 cm^{-1} was recorded, studied and analyzed for the untreated and treated fabric.

X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) patterns onto PP fabric were measured before and after treatment. PP fabric was subjected to XRD with EMPYREAN diffractometer operated at 45KV, (Cu Kα radiation, λ= 1.5406 Å) in 2θ angles ranging from 4.015º to 79.961º with a step size of 0.026º and scanning rate 18.87 seconds.

Antimicrobial activity

PP fabric coated with Cu_2O was analyzed for determination of antimicrobial activity. The fabric was cut and placed within a sterile Petri dish, and 1.0 mL of microorganism was added onto the surface. Fabric was then incubated at 37 °C for 16 h, placed into 10 mL of sterile water and shaken vigorously for 5 min. The solution was serially diluted to 10^4, 10^5, and 10^6concentrations, and 100 µL for each diluted sample was placed onto agar plates. The plates were incubated at 37 °C for 24 h. The antimicrobial behavior of fabric was evaluated quantitatively against the previous coded test organisms. Squares of 1 cm of each fabric were prepared in aseptic manner. Each square was placed in a known concentration of microbial suspension (after calculate colony forming unit (CFU) for this suspension), the reduction in microbial colony (CFU) in standard time was measured. The efficiency of the antimicrobial treatment was determined by comparing the reduction in microbial colony of the treated fabric with that of untreated fabric expressed as a percentage reduction in standard time. The bacteriostatic activity was evaluated after 24h and the percent reduction of bacteria was calculated using the following equation (Eq.3):

$$\%R = [(B - A)/B] \times 100 \quad \text{Eq.3}$$

Where R, the reduction percent, A , the number of bacterial colonies from untreated fabric and B, the numbers of bacterial colonies from treated fabric [28].

Results and Discussion

Synthesis of cuprous oxide micro particles onto PP fabric

In situ synthesis and deposition of cuprous oxide micro particle onto PP fabric was carried out in a bath containing copper sulphate pentahydrate (1-30 mM/L), sodium hydroxide, glucose as reducing agent and PP fabric. Micro cuprous oxide was formed and physically attached to PP fabric (Table1). Scheme1.illustrates the mechanism of copper sulphate reaction with sodium hydroxide and glucose as reducing agent at 90°C for two hrs. After the reduction reaction of copper sulphate in alkali in presence of glucose, PP treated fabric was padded to remove excess solution, dried and cured at 150°C for 5 min. During the thermal treatment $Cu(OH)_2$ was reduced to Cu_2O by action of glucose as reducing agent.

Table 1 shows that the percentage micro Cu_2O deposited onto PP fabric after five washing which increased from 0.18 to 2.16 % using 1-30mM/L $CuSO_4.5H_2O$. The low contents of Cu_2O onto PP are due to its hydrophobic character compared to the highly hydrophilic cellulosic materials.

SEM/EDX

Surface morphology of PP/micro Cu_2O was determined by SEM. In Fig.1 images (a,b,c,d) revealed the synthesis and loading of cuprous oxide micro particles onto PP fabric. Uniformly cubic shaped deposited Cu_2O micro particles of 0.6-0.8 μm in diameter were observed onto PP fabric surface.

$$\underset{\text{Copper sulphate}}{CuSO_4.5H_2O} + 2NaOH \longrightarrow \underset{\text{Copper hydroxide}}{Cu(OH)_2} + Na_2SO_4 + 5H_2O \qquad (a)$$

$$\underset{\text{Copper hydroxide}}{2Cu(OH)_2} + \underset{\text{D-Glucose}}{C_6H_{12}O_6} \xrightarrow{\mathbf{90°C}} \underset{\text{Cuprous oxide}}{Cu_2O} + \underset{\text{D-Gluconic acid}}{C_6H_{12}O_7} + 2H_2O \qquad (b)$$

Scheme 1. Synthesis of cuprous oxide from copper sulphate

TABLE 1. Percentage of Cu_2O microparticles deposited onto PP from $CuSO_4.5H_2O$ in alkaline glucose at 90°C

Wt.$CuSO_4.5H_2O$ gm)	$CuSO_4.5H_2O$ (mM/L)	PP fabric wt (gm)	Fabric wt.after curing (gm)	% Cu_2O deposited on fabric after curing	PP fabric wt. after washing (gm)	% Cu_2O deposited onto PP fabric after washing
0.05	1	1.9004	1.9425	2.2	1.9039	0.184
0.25	5	1.9275	1.9937	3.4	1.9433	0.819
0.75	15	2.1059	2.2110	5	2.1500	2.09
1	20	1.9964	2.1052	5.4	2.029	1.63
1.5	30	2.0126	2.1329	5.9	2.056	2.16

The fabric was analyzed by EDX and show elemental composition of the treated sample (Fig. 2) as follows:

EDX results have confirmed the presence of carbon, copper and oxygen elements onto PP fabric.

Color measurements

The results of color coordinates for the untreated PP and the treated fabric are reported in Table (2). Thus, lightness (L*), redness-greenness (a*), yellowness-blueness (b*) have changed upon the assembling of cuprous oxide micro particles onto PP fabric surface.

All treated samples have acquired a brown brick color. Table 2 shows that the color strength (K/S) increases with the increase of Cu_2O. L* value of the blank is 75.54 and decreases to 47.07 with increase percent Cu_2O, indicating the most significant change which has occurred on the color space. For the treated PP fabric containing Cu_2O, a* and b* values are higher than the untreated fabric. This means a shift of color towards red – yellow area has

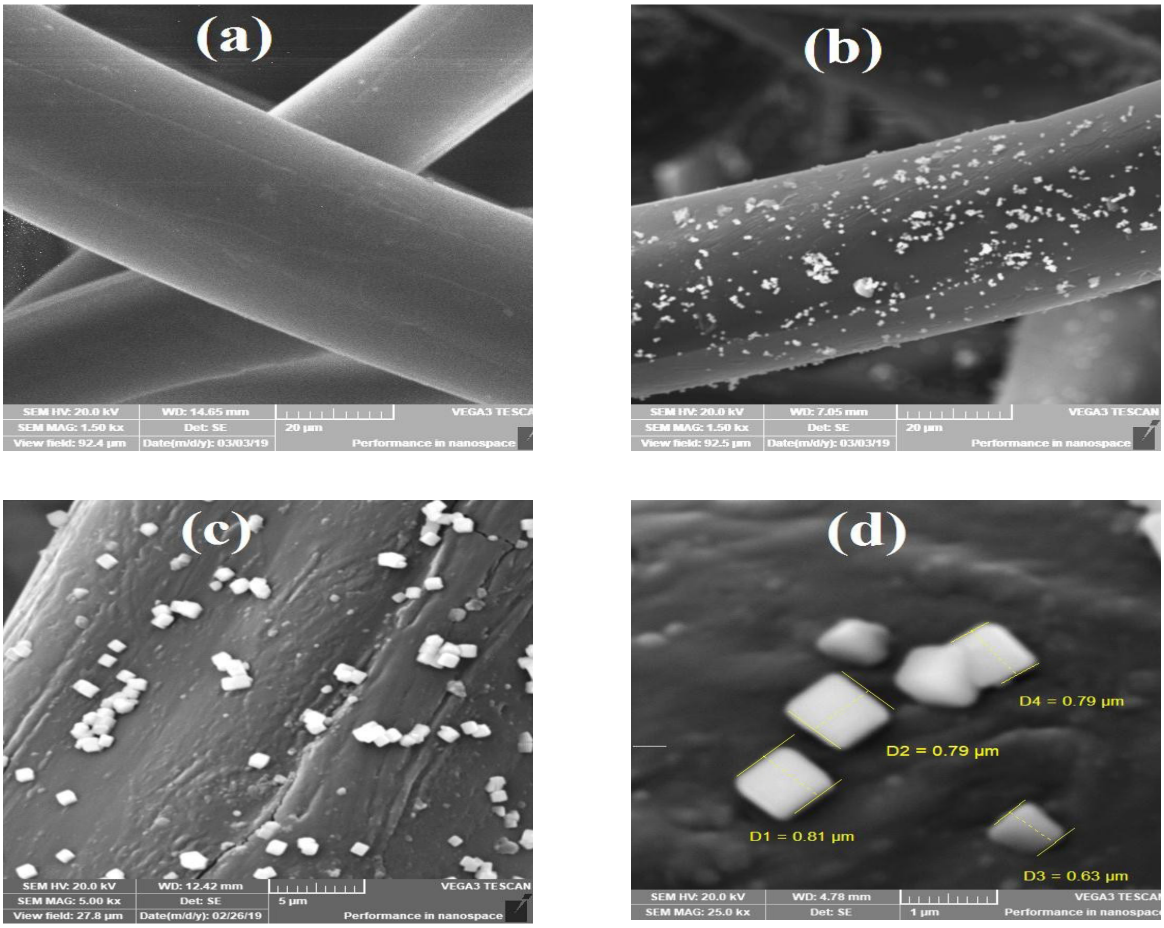

Fig. 1. SEM Images of (a) untreated PP and (b,c,d) PP/micro Cu_2O containing 2.16% Cu_2O.

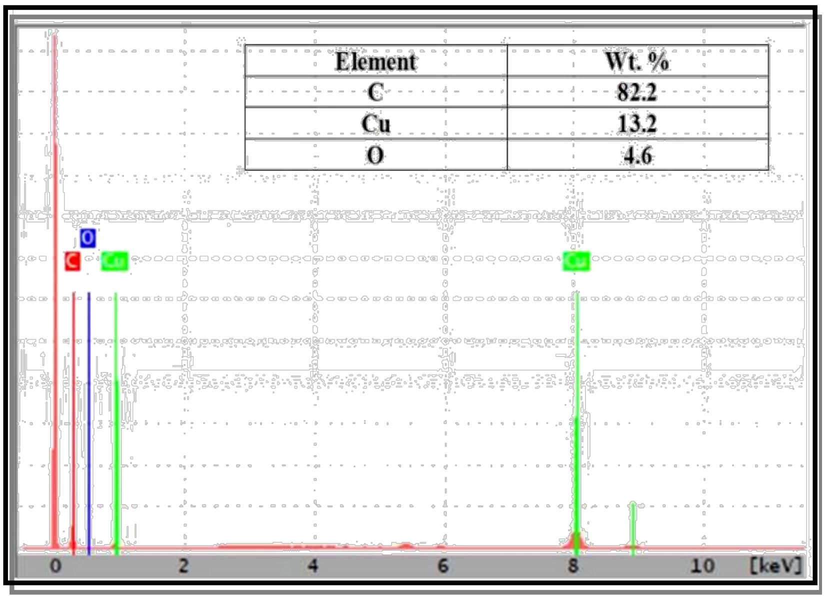

Fig. 2. EDX of PP/ micro Cu_2O.

TABLE 2. Color indices of untreated and treated fabrics at λ_{max}410 nm.

$\%Cu_2O$	K/S	L*	a*	b*	ΔE
PP fabric(blank)	0.36	75.54	-0.15	1.13	0.0
2.09	1.84	54.13	6.15	7.06	22.94
2.16	4.58	47.07	10.93	18.82	36.87

been occurred.

FTIR

Infrared spectra of untreated PP fabric compared to that treated with micro Cu_2O is presented in Fig.3. The absorption band at 628 cm^{-1} indicates the formation of Cu_2O onto PP fabric [29].

XRD

XRD patterns analysis of untreated PP and that coated with PP (2.16 % micro Cu_2O) are demonstrated in Fig. 4. The characteristic peaks at 2θ are 36.5º, 42.4º, 61.5º and 73.7º corresponding to Miller indices (111), (200), (220) and (311), respectively which indicate the presence of Cu_2O with cubic structure onto PP fabric [30].

Antimicrobial Activity

There is an increase demand for antibacterial PP fabrics especially those used in hospitals to prevent infection or transmission of diseases and protect health care workers. In this investigation, PP fabric is bearing chemical and excellent mechanical properties [31] but is lacking antimicrobial properties treated with Cu_2O micro particles as antimicrobial agent, for medical and hygiene applications. The mechanism for the antimicrobial action of copper is known since, copper materials release ions in the presence of water and oxygen, forming complexes with the compounds present in the bacteria. These

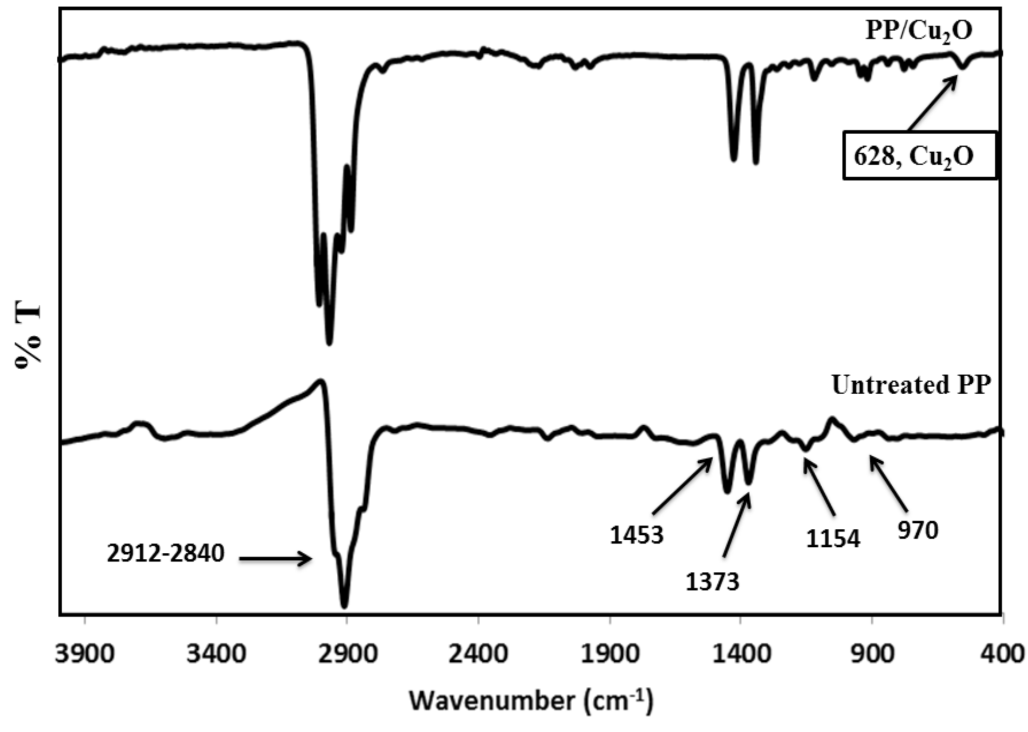

Fig. 3. FTIR of untreated PP and PP/micro Cu_2O fabric containing 2.16% Cu_2O.

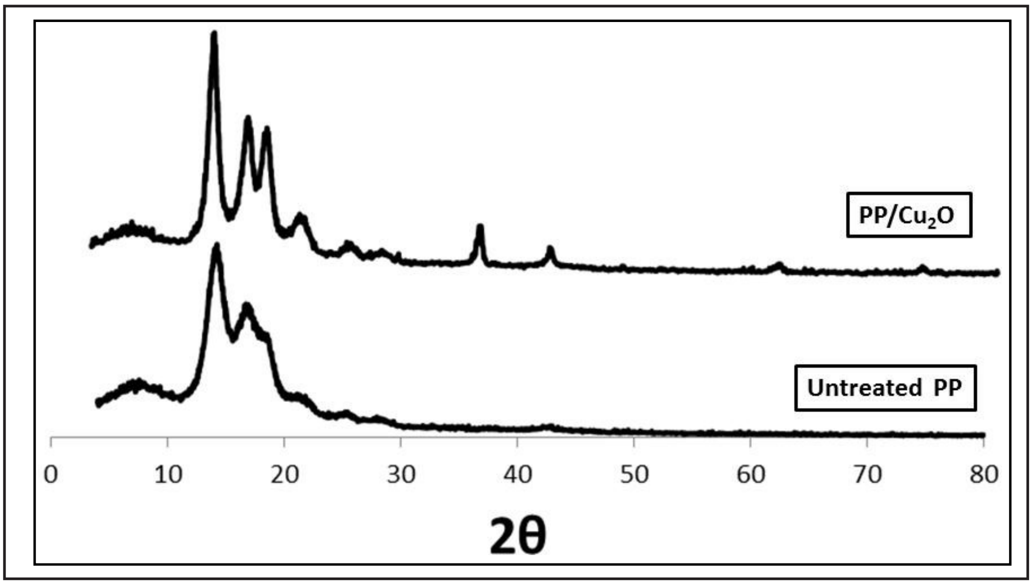

Fig. 4. XRD of untreated PP and PP/micro Cu_2O.

processes may result in damage of the cell wall and proteins, both effects killing the bacteria[32].

Quantitative antimicrobial activity of PP modified by loading Cu_2O micro particles was tested by percentage reduction of bacterial growth using optical density method (OD) against *S. aureus* (gram positive bacteria), *E. coli* (gram negative bacteria) and *C. albicans* (fungi). Table 3 shows the results of percentage reduction growth against the previous organisms which are in the following order:

S. aureus(88%)>*E. coli* (87%)>*C. albicans*(85%).

PP/ Cu_2O nano particles of treated fabric showed maximum percentage reduction for *S. aureus* followed by *E. coli* and then *C. albicans.* They gave very good antimicrobial results.

Also, the enhanced bio-activity of Cu_2O nano particles onto cellulosic materials attained 96.8-97.8 % reduction growth for bacterial and 85.5-89 % for fungi [26]

PP antibacterial fabrics can be used for manufacture of socks, packaging materials, wound healing bandages etc….

Conclusions

In situ synthesis of Cu_2O micro particles onto PP fabric was performed using $CuSO_4.5H_2O$ at 1-30 mM/L, NaOH and glucose as reducing agent at 90ºC for two hours. Then, the fabric was padded, dried, thermally treated at 150ºC for 5 min, washed 5 times, dried and weighed. Cu_2O micro particles were deposited in cubic shape (0.6-0.8µm in diameter) onto PP fabric. They were characterized by XRD, SEM and EDX analysis. Color characterization for untreated and treated PP/Cu_2O micro particles was measured and very good antimicrobial activity was determined. It is an easy synthesis method, of low cost and can be applied in textile industry.

Acknowledgement

The authors would like to thank the central laboratory of textile industries, Research Division of the National Research Center, Cairo, Egypt for the manuscript products analysis.

TABLE 3. Antimicrobial activity of PP fabric loaded with Cu_2O nanoparticles

%Cu_2O	**Percentage reduction of microbial growth**		
	S.aureus	***E.coli***	***C.albicans***
2.16	88	87	85

Funding

This research has not received any specific grant funding from agencies in the public, commercial, or other sectors

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قماش البولى بربيلين المقاوم للميكروبات والمحمل بجزيئات ميكروأكسيد النحاسوز

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في الوقت الحالى زاد الأقبال على مواد النانو تكنولوجى من المعادن وأكاسيدهم لأهميه هذه المواد في أنتاج أقمشه مقاومة للميكروبات.

تم تحضير جزيئات ميكروأكسيد النحاسوز على قماش البولي بروبيلين الغير منسوج وهو يتمتع بخاصية مقاومة الميكروبات بتركيز 30-1 ميلى مول /لتر. وأستعملت الطريقة الكيميائيه بأستخدام كبريتات النحاس المحتويه على خمسه جزيئات من الماء بالأضافة إلى الجليكوز كماده مختزله عند 90°م لمدة ساعتان ثم إزالة السائل الزائد بواسطة الغمر وعصر وتجفيفه، ويلى ذلك تحميص قماش البولى بروبيلين المعالج عند 150°م لمدة خمسه دقائق ثم وزن العينه ثم غسيلها جيدا في الماء عند 50°م لخمسه مرات و تجفيفها وتقدير الزياده فى وزن القماش.

وتم تعريف جزيئات الميكرونحاس فى قماش البولى بروبيلين بواسطة الميكروسكوب الألكترونى و(EDX) وكذلك تشتت أشعة أكس (XRD) وكانت على هيئه مكعبات، وبقطر يتراوح بين 6 .0 – 0.8 ميكرومتر. وتم تقدير تغير لون القماش فى العينات المختلفه عند λ_{max} ، وبدراسه بيانات اللون (*L*, a*, b) مع تغيير شده اللون (K /S). بالأضافه لدراسه القماش المعالج بمادة Cu_2O والمقاوم للميكروبات وأثبتت التجارب انه ذات مقاومه جيده جدا للميكروبات والفطريات.