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Multifunctional Nonwoven Polypropylene Fabric loaded with (Cu_2O/Ag) Nanocomposites for Potential Industrial Applications

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Abstract

Nanotechnology is continuously employed to produce textiles with functional properties such as antimicrobial activity and ultraviolet (UV) protection. Cuprous oxide/silver (Cu_2O/Ag) nanocomposites were in situ prepared onto nonwoven PP fabric by chemical reduction method. This method is in good agreement with ecofriendly, simplicity and cost-effectiveness concepts. The morphology of PP/Cu_2O/Ag nanocomposites was observed by scanning electron microscope (SEM). The obtained PP nanocomposites were characterized using energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD). Antibacterial activity of PP/Cu_2O/Ag nanocomposites were evaluated against *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) bacteria. UV protection factor (UPF) and electrical conductivity were measured. Thermal properties including thermogravimetric analysis TGA, derivative thermogravimetry (DTG) and differential scanning calorimetry (DSC) were studied. Color properties were determined before and after treatment. The obtained results of SEM, EDX and XRD clearly emphasized the successful formation and incorporation of Cu_2O/Ag NPs onto the surface of PP fibers. It is found that PP/Cu_2O/Ag nanocomposites have excellent bactericidal effect and UV protection. The treatment has not significantly affect the thermal properties of PP. Furthermore, electrical conductivity of PP/Cu_2O/Ag nanocomposites has slightly increased compared to untreated PP fabric. The prepared PP/Cu_2O/Ag nanocomposites with multifunctional properties can be useful in developing different industrial products, for example, filters, geotextiles, automotive, medical and hygienic products, etc.

Keywords: Nonwoven polypropylene fabric; multifunctional properties; antibacterial activity; UPF; electrical conductivity; coloration

1. Introduction

Polypropylene (PP) is distinguished by its low production cost, lightness, and good chemical, physical and mechanical properties [1, 2]. Therefore, it is used as fibers, woven and nonwoven fabrics in different industrial applications such as indoor/outdoor carpets, automotive and upholstery fabrics. Nonwoven PP fabrics are widely utilized in geotextiles, purification filters, medical and hygienic products [3-10]. Nevertheless, PP is known for its hydrophobicity, low electrical conductivity and poor UV protection. Consequently, several surface modification techniques are adopted to improve PP properties and add new functionalities such as grafting, plasma pretreatment and melt spinning. Several additives and substances are used to functionalize PP, e.g. nanoparticles (NPs) of metal and metal oxides. They are applied to provide PP with antimicrobial activity, protection from UV radiation, photocatalytic property, etc [11-18]. The degradation of PP fibers induced by UV radiation affects their use in many outdoor applications. As they are well known for their photocatalytic effect, Zinc oxide (ZnO) and titanium dioxide (TiO_2) NPs were utilized to acquire PP with UV shielding effect. PP fibers were firstly coated with polydopamine layer, then TiO_2 NPs were chemically bound to the coated fibers [19]. Sinha et al., used electrospinning to fabricate nanoweb of PP coated with polyvinylidene difluoride followed by functionalization with TiO_2 NPs [20]. Nonwoven PP fabric with better UV protection were prepared using dielectric-barrier discharge (DBD) plasma. Then, ZnO and TiO_2 NPs were loaded onto activated PP fabrics [21]. Feng et al., prepared PP fibers with ZnO/TiO_2 nanocomposite by melt blending technique. They found that PP nanocomposites showed outstanding UV shielding [22]. PP fibers and fabrics with

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antimicrobial and photocatalytic functionality were produced using different nanofillers such as Ag, ZnO, Zn [23-26]. Antibacterial PP fabrics functionalized with Ag NPs were used as the core for fabricating wound healing dressing and face masks [27, 28]. Cu/Ag thin films were deposited onto PP fibers using the magnetron sputtering technique. Cu/Ag nanocomposite film enhanced abrasion resistance, antibacterial and electrostatic properties of PP fibers [29]. Nonwoven PP fabrics loaded with Ag and Ag/Cu NPs have effectively applied for reducing 4-nitrophenol due to the catalytic efficiency of Ag and Cu NPs. The prepared PP nanocomposites were suitable for the treatment of waste water [30, 31].

Cuprous Oxide (Cu_2O) has gained much interest as it is a nontoxic p-type semiconductor that has magnetic, electronic and optical properties. It can be prepared by different methods including thermal oxidation, spray pyrolysis and chemical reduction. The later method is preferred because it is simple and inexpensive. The common reducing reagents are sodium citrate, L-ascorbic acid and sodium borohydride. Also, natural reducing agents are used e.g. liginin, glucose, starch or plant extracts [32-34]. Cu_2O micro particles with different structures (cube, needle) were simultaneously synthesized and deposited onto different fabrics; PP, viscose and cotton, respectively. These treated fabrics showed excellent microbial reduction [35-37].

This work aims to fabricate PP/Cu_2O/Ag nanocomposites by simple and ecofriendly technique. The prepared PP nanocomposites were characterized using SEM, EDX XRD in addition to TGA, DTG and DSC. Antibacterial activity of PP/Cu_2O/Ag nanocomposites against pathogenic microbes (*S. aureus and E. coli*) was evaluated. UPF, electrical conductivity and color properties were assessed before and after treatment.

2. Materials and methods

2.1. Materials

Nonwoven PP fabric (200 g/m^2, thickness 2.60 mm) was supplied by Egyptex Co., Cairo. The fabric was well washed with acetone before treatment to remove lubricants and impurities adsorbed on the surface, as reported in the literature [38, 39]. Copper sulphate pentahydrate ($CuSO_4.5H_2O$), silver nitrate ($AgNO_3$), sodium hydroxide (NaOH) and D-glucose ($C_6H_{12}O_6$) were bought from local market and used without further purification.

2.2. Preparation of polypropylene/cuprous oxide/silver nanocomposites (PP/Cu_2O/Ag)

In situ synthesis of mixtures of Cu_2O/Ag NPs onto nonwoven PP fabrics was performed by immersing the fabrics (10 cm x 10 cm) in a solution containing mixture of $CuSO_4.5H_2O$ (30 mM) and $AgNO_3$ (30 mM) with three different mixing ratios [$CuSO_4.5H_2O$: $AgNO_3$, (75:25, 50:50 and 25:75)] and liquor ratio 1:100. Under continued shaking, NaOH (100 mM) was added drop by drop to the reaction solution at 60 °C followed by the addition of D-glucose (25 mM). The temperature was raised to 90 °C and maintained at that temperature for two hours. The fabrics were padded to 100 % (wt/wt) wet pick up using a padder (Roaches Co, England), dried at 100 °C for one hour and cured at 150 °C for 5 min. The treated fabrics were rinsed 5 times with distilled water for 10 min and dried at 100 °C.

2.3. Analysis and Measurements

2.3.1. Morphological studies by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX)

The morphology of nonwoven PP fabrics was observed by high resolution electron microscope (SEM Tescan Vega 3 SBU) working at 20 KV. EDX analysis was carried out to detect and confirm the elemental composition of the samples.

2.3.2. X-ray diffraction (XRD)

X-ray diffraction (XRD) patterns of nonwoven PP fabrics were detected before and after treatment. Fabrics were subjected to EMPYREAN diffractometer system operated at 45KV, (Cu Kα radiation, λ= 1.5406 Å) in 2θ angles ranging from 5º to 80º with a step size of 0.026º and scanning rate 21.42 seconds.

2.3.3. Antibacterial activity test

Antibacterial activity of the treated nonwoven PP fabrics was assessed according to standard methods AATCC TM100. It was performed as follows: 4 ml sterile nutrient broth was inoculated overnight with subcultures of the used microorganisms [gram positive (*S. aureus*) and gram negative (*E. coli*)] separately to prepare final count 10^4 CFU/ml. 100 mg of samples (after exposure to UV for 20 min) were added to the inoculated broth and incubated at 37 °C for 24 h (Sample). Another 4 ml sterile nutrient broth was used as blank after addition of the same sample weight (100 mg) without addition of microorganisms. The positive controls contained nutrient broth and microorganisms only. Another 4 ml sterile nutrient broth was used as negative control without addition of sample neither microorganisms. After incubation of all samples, blank, positive and negative controls at the same conditions, optical density (OD) was measured at wavelength (630 nm). The OD readings for samples were measured against their blanks and the positive control OD readings were measured against the negative control. Bacterial reduction percentages were determined from equation 1:

$$Reduction\ (\%) = \frac{Positive\ control\ OD - Sample\ OD}{Positive\ control\ OD} \times 100 \qquad \text{(Eq.1)}$$

2.3.4. Ultraviolet protection factor (UPF)

The ability of fabrics to block UV light is expressed by the ultraviolet protection factor (UPF). The measurement of UPF value was performed according to the Australian/New Zealand standard method (AS/NZS 4399:1996) [40] using UV/Visible Spectrophotometer JASCO V-750 (from 280-400 nm at an interval of 5 nm). Equation 2 is used to estimate the UPF value as follows:

$$UPF = \frac{\sum_{290}^{400} E_\lambda \times S_\lambda \times \Delta\lambda}{\sum_{290}^{400} E_\lambda \times S_\lambda \times T_\lambda \times \Delta\lambda} \qquad \text{(Eq. 2)}$$

Where; E_λ is the relative erythemal spectral effectiveness, S_λ is the solar spectral irradiance in $W.m^{-2}.nm^{-1}$, T_λ is the spectral transmittance of the item, $\Delta\lambda$ is the wavelength step in nm and λ is the wavelength in nm.

2.3.5. Thermal Properties

Thermal behavior of untreated and treated PP fabrics was analyzed by TGA, DTG and DSC. These analyses were performed using SDT Q600 V20.9 Build 20. Samples were heated at the rate of 10 ˚C/min from room temperature to 800 ˚C in nitrogen atmosphere.

2.3.6. Electrical conductivity measurement

Electrical conductivity (σ) of PP fabrics was measured using Computerized LRC-bridge (Hioki model 3531 zHi Tester). PP samples were studied at room temperature and frequencies ranging from (100 Hz up to 100 KHz). The studied samples were 10 mm in diameter. The electric measurements were carried out by inserting the sample between two parallel plate conductors forming cell capacitor. The conductivity was calculated from equation 3:

$$\sigma = 2\pi f d C \tan\delta / A \qquad \text{(Eq. 3)}$$

Where σ: A.C. conductivity, f: operating frequency, d: thickness of the dielectrics, C: capacitance, tan δ: dielectric loss and A: area of the electrode.

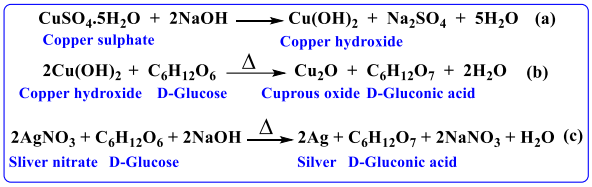
2.3.7.Color properties measurement

Color strength (K/S) and coordinates (L*, a* and b*) of CIE LAB color system for PP fabrics were obtained using Ultrascan Pro Hunter Lab. L* indicates the lightness/darkness (from 100 to 0), a* values refer to the redness (positive) – greenness (negative) and b* values represent yellowness (positive) – blueness (negative).

3. Results and Discussion

3.1. Preparation of polypropylene/cuprous oxide/silver nanocomposites (PP/Cu_2O/Ag)

Multifunctional nonwoven PP fabric was prepared by loading mixtures of Cu_2O/Ag NPs onto its surface. Cu_2O and Ag NPs were in situ synthesized by reducing $CuSO_4.5H_2O$ and $AgNO_3$ with glucose in basic medium. Glucose was chosen because it is natural, abundant and strong reducing agent. The reduction mechanism of both $CuSO_4.5H_2O$ and $AgNO_3$ using glucose is demonstrated in Scheme 1. Solutions of $CuSO_4.5H_2O$ and $AgNO_3$ were mixed with three ratios (75:25, 50:50 and 25:75), respectively. The properties of the prepared PP/Cu_2O/Ag nanocomposites were studied and compared with the untreated PP.



Scheme 1. Synthesis of Cu_2O and Ag by chemical reduction of $CuSO_4.5H_2O$ and $AgNO_3$

3.2. Morphological studies by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX)

Surface morphology of untreated and treated nonwoven PP fabrics was observed by SEM and presented in Fig. 1 (a-d). SEM micrographs were recorded at two magnifications: 1.5 kx and 30 kx. As shown in Fig. 1a, the untreated PP fabric has a smooth surface and uniform morphology. After the deposition of Cu_2O/Ag NPs, the morphology of PP fabric is changed to a rough and non-homogenous surface as demonstrated in Fig. 1(b-d). It can be seen that the surface of PP fabrics is coated with both Cu_2O and Ag NPs in the form of cubes and spheres, respectively. The formed particles have average particle size of approximately 200 nm for Cu_2O and < 100 nm for Ag.

PP fabrics were characterized by EDX and their spectra are demonstrated in Fig. 2 (a-d). EDX spectrum recorded for the untreated PP fabric (Fig. 2a) revealed that it is mainly consist of carbon. Fig. 2 (b-d) represents EDX spectra of PP fabrics loaded with mixtures of Cu_2O/Ag NPs and the peaks relative to copper, oxygen, silver as well as carbon are all found. The mentioned findings indicate the synthesis and incorporation of Cu_2O/Ag NPs onto nonwoven PP fabrics.

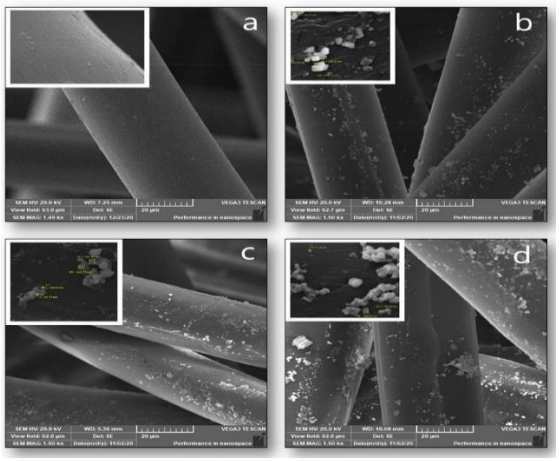


Fig 1. SEM photos at MAG: 1.5 kx and MAG: 30 kx at the upper left side [a) untreated PP, b) PP/ Cu_2O/Ag (75:25), c) PP/ Cu_2O/Ag (50:50), d) PP/ Cu_2O/Ag (25:75)]

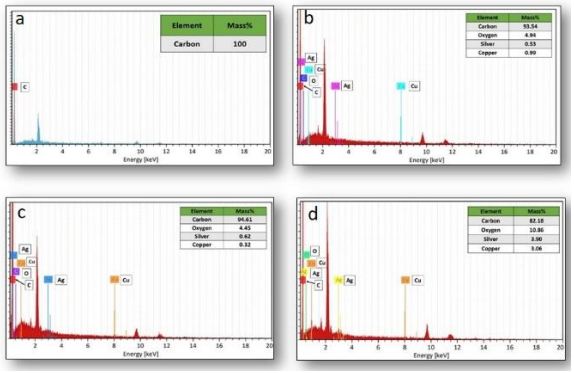


Fig 2. EDX spectra [a) untreated PP, b) PP/ Cu_2O/Ag (75:25), c) PP/ Cu_2O/Ag (50:50), d) PP/ Cu_2O/Ag (25:75)]

3.3. X-ray diffraction (XRD)

PP fabrics were scanned by XRD diffractometer in 2θ angles ranging from 5° to 80°. XRD spectra of untreated and treated PP fabrics are displayed in Fig. 3 (a-d). The typical XRD pattern for PP is depicted in Fig. 3a, there are four diffraction peaks at 2θ [14.2°, 16.8°, 18.5° and 21.4°] corresponding to Miller indices [(110), (040), (130) and (131/041)] planes of polypropylene, respectively [41]. Fig. 3(b-d) presents the XRD patterns for PP fabric loaded with Cu_2O/Ag NPs. For sample PP/Cu_2O/Ag (75-25), the diffraction peaks corresponding to face centered cubic Cu_2O can be observed at 2θ [36.5° (111) as well as weak and broad peaks at 2θ [42.4° (200) and 61.5° (220)] (JCPDS: 05-0667) [35, 42] in addition to the peaks of Ag at 2θ [38.6° (111) and 77.7° (311)] [43]. By increasing the concentration of $AgNO_3$ in the mixture, the intensity of the characteristic peak for Cu_2O at 2θ [36.5° (111)] diminished while that for Ag at 2θ [38.6° (111)] intensified. The diffraction peaks at 2θ [38.6° (111)] noticed with small and wide peaks at 2θ [44.5° (200), 64° (220) and 77.8° (311)] refers to the existence of face centered cubic Ag (JCPDS: 04-0783) and indicates that more Ag NPs were deposited onto PP surface (Fig. 3d) [44].

The crystallinity index (CI) was determined to examine the influence of treatment on the crystallinity of PP fabric and it is demonstrated in Fig. 3 (a-d). The crystallinity index of all PP nanocomposites is less than untreated PP. This decrease refers to the increase in the amorphous region in PP due to the deposition of Cu_2O/Ag NPs on the surface of fabric and between the fibers.

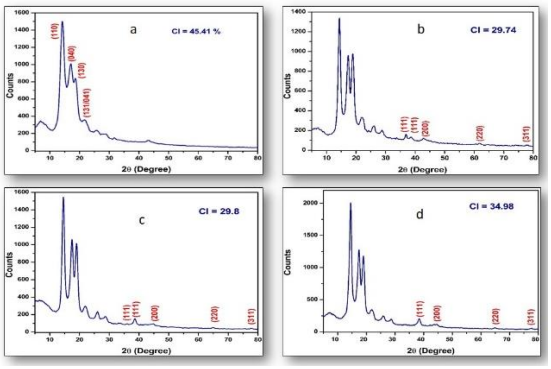


Fig 3. XRD patterns [a) untreated PP, b) PP/ Cu_2O/Ag (75:25), c) PP/ Cu_2O/Ag (50:50), d) PP/ Cu_2O/Ag (25:75)]

3.4. Antibacterial activity

Many researchers investigated the potential application of Cu_2O and Ag NPs as antibacterial finishing for various fabrics and textiles [45-48].

The antibacterial activities of untreated PP fabric and the synthesized PP/Cu_2O/Ag nanocomposites were examined against *S. aureus* as gram-positive bacteria and *E. coli* as gram-negative bacteria. The results of the reduction in bacterial growth are displayed in Fig. 4. The synthesized PP/Cu_2O/Ag nanocomposites exhibited remarkable bacteriostatic activity against both *S. aureus* and *E. coli*. These bacteriostatic activities can be related to the loading of Cu_2O/Ag NPs onto PP fabric.

Yang et al., investigated the antibacterial activity of Cu_2O/Ag nanocomposite against gram-positive bacteria (*S. aureus*) and gram-negative bacteria (*P. aeruginosa*). They found that Cu_2O/Ag nanocomposite showed long-term sterilization activity against both pathogens. Additionally, they suggested a possible mechanism for the effect of Cu_2O/Ag nanocomposites on the tested bacteria. They indicated that the photoexcited electrons and holes produced from Cu_2O/Ag composite attacked bacteria and induced the excess accumulation of intracellular reactive oxygen species (ROS) such as H_2O_2, superoxide anions ($\cdot O_2^-$) or hydroxyl radicals ($\cdot OH$). The active ROS induced nucleic acids damage, intracellular protein inactivation, dysfunction of the mitochondria, and the gradual disintegration of the cell membrane leading to cell death [49].

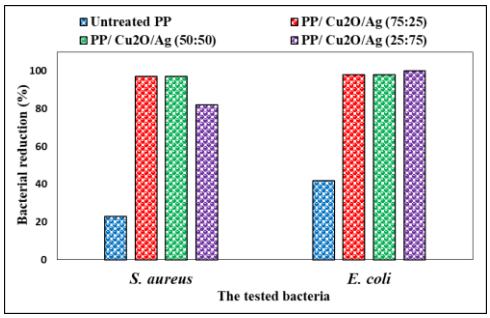


Fig 4. Antibacterial activity of untreated and treated PP fabrics

3.5. Ultraviolet protection factor (UPF)

PP is liable to chain degradation from exposure to UV radiation. Additives such as UV absorbers or blockers are used to maintain PP stability and preserve its physical and mechanical properties. Nanoparticles are used to acquire fabrics with UV protection. It was reported that Cu_2O and Ag were used to enhance the UV shielding of natural and synthetic fabrics [45, 50].

UPF is used to evaluate the UV shielding performance of fabric products. UPF was determined after imparting Cu_2O/Ag NPs on PP fabric and the results were compared with the untreated fabric. As demonstrated in Table 1, untreated PP fabric has inadequate protection against UV radiation (UPF: 1.3). The obtained PP/Cu_2O/Ag nanocomposites possessed excellent UV protection. It can be noticed that increasing the concentration of Cu_2O NPs in the mixture lead to enhancing the UPF value from 42.7 for [sample: PP/ Cu_2O/Ag (25:75)] to 328.9 for [sample: PP/ Cu_2O/Ag (75:25)].

Table 1. UPF values for untreated and treated PP fabrics

Sample	UPF values	Protection category*
Untreated PP	1.3	Insufficient
PP/ Cu_2O/Ag (75:25)	328.9 (50+)	Excellent
PP/ Cu_2O/Ag (50:50)	78.1 (50+)	Excellent
PP/ Cu_2O/Ag (25:75)	42.7	Excellent

*UPF protection category according to AS/NZS 4399:1996 (<15: insufficient protection, <15-24>: good, <25-39>: very good, > 40-50, 50+: excellent)

3.6. Thermal properties

TGA curves of untreated and treated PP fabrics are displayed in Fig. 5. It can be noticed that the obtained PP/Cu_2O/Ag nanocomposites exhibited similar decomposition pattern as the untreated PP. Also, the thermal decomposition of all PP samples occurred in a single step.

DTG profiles for all samples are illustrated in Fig. 6. The thermal decomposition of untreated PP began at a temperature of 397 ºC and the maximum decomposition temperature occurred at 462 ºC. This temperature represents the decomposition of the carbon content of PP. The treated PP samples showed higher maximum decomposition temperature (466 ºC) compared to untreated PP. It could be concluded that the incorporation of Cu_2O/Ag NPs slightly improved the thermal stability of PP.

DSC thermographs are demonstrated in Fig. 7 and there is an endothermic peak at 172 °C corresponds to the melting temperature (T_m) of PP. The melting temperature of PP has shifted to lower temperatures (168-170 °C) after the addition of Cu_2O/Ag NPs. The decrease in the melting point indicates that the crystalline phase of PP has changed after the deposition of Cu_2O/Ag NPs.

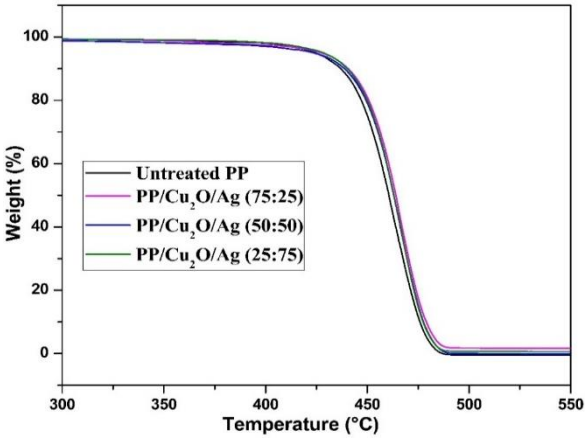


Fig 5. TGA curves of untreated and treated PP fabrics

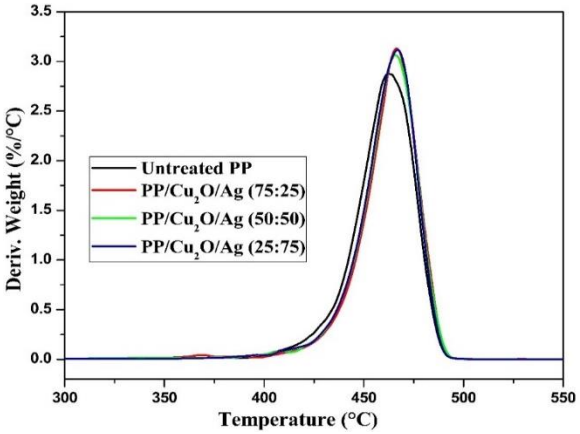


Fig 6. DTG profiles of untreated and treated PP fabrics

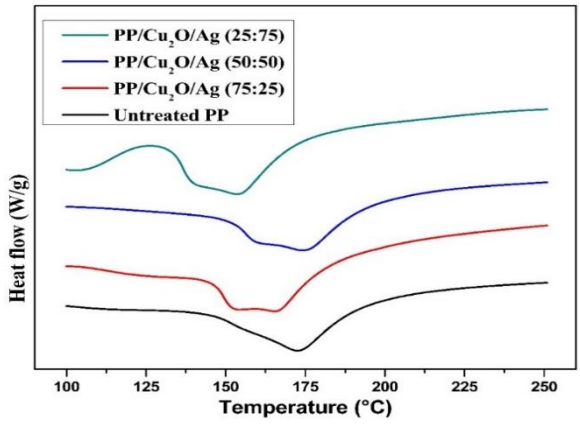


Fig 7. DSC thermographs of untreated and treated PP fabrics

3.7. Electrical conductivity measurement

Electrical conductivity of PP/Cu_2O/Ag nanocomposites in addition to the untreated fabric are displayed in Fig. 8. The synthesized PP nanocomposites exhibit slight increase in electrical conductivity compared to untreated PP. The synthesized PP nanocomposites exhibit slight increase in electrical conductivity compared to untreated PP. PP/Cu_2O/Ag (50:50) nanocomposite showed the higher improvement in electrical conductivity compared to the other prepared PP/Cu_2O/Ag nanocomposites. This is could be attributed to the presence of the conductive metal oxide/metal (Cu_2O/Ag NPs) loaded onto PP surface. Moreover, Montazer et al., reported that the conduction occurs when the distances between the conductive particles are close enough to form a continuous network of charge carriers [51].

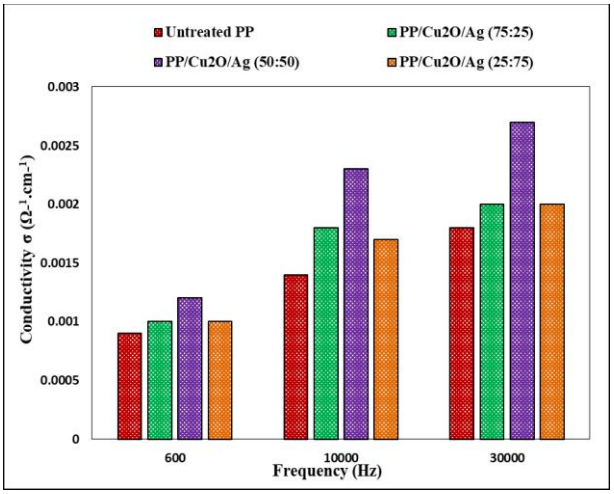


Fig 8. Electrical conductivity for untreated and treated PP fabrics

3.8. Color properties measurement

In situ synthesis of Cu_2O /Ag nanoparticles on fabrics considerably affected the color of PP fabric as shown in Fig. 9. The treatment, also, changed the color coordinates (L*, a* and b*) as demonstrated in Table 2. The color shade varies according to the shape of particles and the deposited percentage of each nanoparticles and this depend on nonhomogeneous density of the used nonwoven PP fabric [45, 47, 48]. The highest K/S value was noticed for sample PP/Cu_2O/Ag (50:50). L* measured for PP/Cu_2O/Ag nanocomposites reduced to lower values which indicate that the color of PP fabric became darker after the treatment. The values of a* and b* for the obtained PP nanocomposites are shifted towards the red yellowish zone of CIE lab color space.

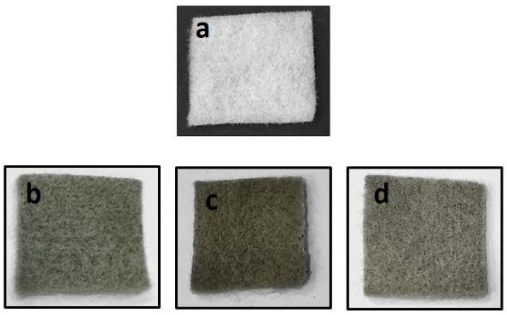


Fig 9. Photos of untreated and treated PP fabrics; [a) untreated PP, b) PP/ Cu_2O/Ag (75:25), c) PP/ Cu_2O/Ag (50:50), d) PP/ Cu_2O/Ag (25:75)]

Table 2. Color indices of untreated and treated PP fabrics

sample	λ_{max} (nm)	K/S	L*	a*	b*
Untreated PP	-	-	75.54	-0.15	1.13
PP/ Cu_2O/Ag (75:25)	375	4.68	42.91	2.59	8.05
PP/ Cu_2O/Ag (50:50)	380	5.03	39.70	4.3	4.45
PP/ Cu_2O/Ag (25:75)	405	2.88	48.01	1.02	3.51

4. Conclusion

Fabrication of multifunctional PP was carried out by ecofriendly, simple and inexpensive procedure. Nonwoven PP fabric was modified by in situ incorporation of Cu_2O/Ag NPs on its surface. Untreated and treated PP fabrics were characterized by SEM, EDX and XRD. The results confirm the synthesis and loading of Cu_2O/Ag NPs onto the surface of the nonwoven PP fabrics. The treated fabrics showed outstanding antibacterial activity against *S. aureus* and *E. coli* and possessed superior UV protection with good electrical conductivity. The thermal analysis revealed that all samples have higher thermal stability than the untreated PP. Moreover, the synthesized PP nanocomposites have gained different colors after deposition of Cu_2O/Ag NPs. Accordingly, the multifunctional PP loaded with Cu_2O/Ag NPs can be applied in many industrial applications such as filters, carpets, automotive, geotextiles, medical and hygienic products, etc.

Conflict of interest

The authors have no conflict of interest to declare.

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