FLAME RETARDANT AND UV-PROTECTIVE COTTON FABRICS FUNCTIONALIZED WITH COPPER(II) OXIDE NANOPARTICLES

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ABSTRACT

Burn and Ultraviolet (UV) Radiation injury has been one of the threats in the military environment. As the rate of death cases caused by burning and skin diseases dramatically increases, the researchers seek a nanocomposite that can improve the properties of the cotton fabrics such as flame retardant and UV-protective properties. For the enhancement of the functional properties of fabrics, the use of nanotechnology has been one of the solutions. Therefore, the raw material copper (II) chloride was subjected to the synthesis of CuO nanomaterial by chemical precipitation method. The copper (II) oxide was crystallized and became a nanoparticle by annealing it at temperature 200˚C and 600˚C respectively. Cotton fabrics were separately functionalized with CuO (bulk), CuO-NPs (200˚C), and CuO-NPs (600˚C) by an optimized pad-dry-cure method. The fabrics undergo a flame retardancy test and revealed that CuO (bulk) has a significant flame-retardant property. CuO-NPs (200˚C) treated fabric possesses higher flame retardancy and CuO-NPs (600˚C) fabric being the highest. The fabrics were also used for UV analyses to determine their UV-protective properties. The tests stated that CuO has excellent UV-protection. However, the CuO-NPs (200˚C) fabric has higher UV-protection properties and highest in CuO-NPs (600˚C) treated fabric. The nanoparticle annealed at the highest temperature (600˚C) produced the smallest size of nanoparticle which made the CuO attach well to the surface of the fabrics and provide higher functional properties. This study proved that CuO-NPs can be a promising finishing agent for textile materials and be used for consumer and military applications.

Keywords: copper(II) oxide (CuO), copper(II) oxide nanoparticles (CuO-NPs), annealing temperature, functional properties, flame retardancy, Ultraviolet Protection Factor (UPF), Ultraviolet A-rays (UVA) Transmission, Ultraviolet B-rays (UVB) Transmission, cotton fabrics

INTRODUCTION

There are an estimated 180 000 death cases annually caused by burns according to the World Health Organization. Consumers and fire safety organizations are having critical observations on clothing that can erupt into flames. They stated that Federal regulations governing the safety of the fabrics used in military clothing are too weak and vulnerable to different kinds of hazards during combat (Kerr, 1983). Textiles were improved to protect against environmental influences in any circumstances. Textile garments typically have protective properties against the effects of Ultraviolet (UV) Radiations but are only limited. Clothing textiles that are being worn nowadays have insufficient protection such as flame retardancy and UV-protective. Most affected people were concerned, which is the military (Stephens, Herndon, Colón & Gottschalk, 2011).

Recently, textile industries have given their attention to the improvement of the functional properties of fabrics. Cotton fabrics available in the market have low durability, very combustible, and do not contain enough UV-protection. These concerns became very alarming and became a challenge to fabric manufacturers on improving their textile globally. The circumstances became a challenge that a lot of manufacturers are facing nowadays. Nanotechnology, a new method of improving fabrics with enhanced functional properties became a trend for textile industries. The market in military fabrics perceives and demands a better quality of cotton fabrics used for military clothing in combats since this population is commonly exposed in a hazardous combat environment (Przybylak, Maciejewski, Dutkiewicz, Wesołek & Władyka-Przybylak 2015).

Textiles utilize the metal oxides/ceramics that were synthesized into nanoparticles to provide sufficient functional properties into textiles. Metal oxides that were commonly used in a textile that provides fabrics functional properties are titanium oxide (TiO_2), zinc oxide (ZnO), and magnesium oxide (MgO). There are only a few studies about copper (II) oxide as a textile coating (Alongi, Ciobanu & Malucelli, 2012).

This study assesses the copper (II) oxide nanoparticles as finishing nanosol for cotton fabrics. The functional properties of coated fabrics were evaluated. Specifically, this study was conducted to determine the flame retardant and UV-protective properties of cotton fabrics functionalized with copper (II) oxide nanoparticles (CuO-NPs).

LITERATURE REVIEW

Functional Properties of Metal Oxides Nanoparticles on Fabrics

The flexibility of the cotton fabrics has been popular because of their low cost, biodegradability, and softness. However, even though this kind of fabric has a wide application, these fabrics still have an insufficient flame and UV protection. It led to using metal oxides as a trend on improving textile qualities. Recently, the use of nanotechnology in textiles is dramatically increasing because the surface of the fabrics can effectively utilize the functional properties of the metal oxides (Dhineshbabu & Bose, 2018). Some of these functional properties are flame retardancy and UV-protective.

Flame Retardancy

The term flame retardant from the word itself 'retardant' refers to protection from an outbreak of fire on the surface of the fabrics. In the study of Rajendran, Dhineshbabu, Kanna, and Kaler (2014), the researchers used a different kind of metal oxide which is MgO or known as magnesium oxide. Magnesium oxide is a non-harmful white powder that is constantly utilized in industrial applications. It is said that it is one of the most useful metal oxides when improving the functional properties of fabrics especially its fire retardancy. They claimed that fabric coated with the nanosol prepared using magnesium oxide and methyl silicate has higher flame retardancy than the fabric coated with methyl silicate fabric because of the presence of a metal oxide.

Another study was made (Rajendran et al. 2014) to investigate the flame retardancy of metal and metal oxides by using ZrO_2, MgO, TiO_2 as coatings on fabrics. This study was made because of the trend on the use of metal nanoparticles such as Ag, Au, and graphene. There is also a trend on the use of metal oxides such as zinc oxide, titanium oxide, and others to treat fabrics. In this study, three metal oxides were nanosized and used as a treatment for fabrics. The results revealed that the application of the three metal oxides improved the flame retardancy and thermal stability of the fabrics. Rajendran et al. (2014) concluded that metal oxide nanoparticles are effective to be used as finishing for textiles.

UV Resistance

High Ultraviolet Protection Factor (UPF) on fabrics are very essential for people. As people go out every day, they do not mind about blocking the transmission of UV Radiations from the sun. However, having treated fabrics that can provide UV protection is a huge difference. A study conducted by Kathirvelu, D'Souza, and Dhurai (2008) indicate that the use of metal oxide nanoparticles such as TiO_2-NPs and ZnO-NPs on fabrics are effective to be UV absorbers. The UV tests of this study proved that metal oxide nanoparticles have significant UV-protective properties.

Functional Properties of Copper (II) Oxide Nanoparticles on Fabrics

Copper (II) oxide is one of the metal oxides that contain properties like flame resistance and protection from UV rays. Copper is a significant metal that is frequently used in many areas because of its thermal conductivity, and it has great antibacterial activity towards pathogens (Suryaprabha & Sethuraman 2016).

In another study of UV-protective fabrics using copper nanoparticles, cotton fabrics that are deposited with nanometal oxide films showed excellent and enhanced protection against ultraviolet radiation. The use of UV-protective and flame-retardant fabrics in the form of medical clothes, protective garments, and bedspreads is a growing awareness towards people to minimize the chance of nosocomial infections (El-Nahhal et al. 2012). Copper (II) oxide nanoparticles were stored into cotton fibers through the process of ultrasonic irradiation. It was claimed that the chemical conditions and physical environment will affect the growing process of the particles created onto the cotton fibers. These materials can be used as multifunctional fabrics such as medical cloth, personal protective equipment (PPE) and bedspreads, and many other ways to minimize the chance of hospital-acquired infections. Copper (II) oxide will be very effective in shielding Ultraviolet Radiations because it is used as a nanoparticle.

Hypotheses

H1: The greater the temperature exposed to copper (II) oxide, the smaller size it will become.

H2: The smaller the size of the copper (II) oxide synthesized by precipitation transformation is, the higher its attachment and treatment to the surface of the fabrics.

H3: CuO-NPs have significant flame retardant and UV-protective properties.

H4: CuO-NPs can be a finishing agent for cotton fabrics.

METHODS

Materials

Copper (II) chloride for the synthesis of copper (II) oxide nanoparticles was gathered from ESME Organics. Bulk copper (II) oxide was gathered from Puljed Trading Laboratory and Medical Supplies. Bleached and scoured cotton fabrics were used as a substrate. The fabric was 8.27 × 11.69 inches each. Tetraethyl orthosilicate (TEOS), sodium hydroxide, ammonia, ethanol, and ethylene glycol, all from PTRI - DOST and deionized water were used.

Sample

To determine the multifunctional properties of cotton fabrics treated with CuO-NPs nanocomposites, the researchers prepared four samples of 8.27 in × 11.69 in cotton fabrics treated with different solutions. The first sample is the negative-control set-up which is the uncoated fabric. The second sample is the cotton fabric treated with bulk copper(II) oxide, and the positive control set-ups would be the fabric treated with silica nanosol prepared by CuO-NPs exposed in 200oC and 600oC temperature during the synthesis of nanoparticles.

Data Gathering Procedure

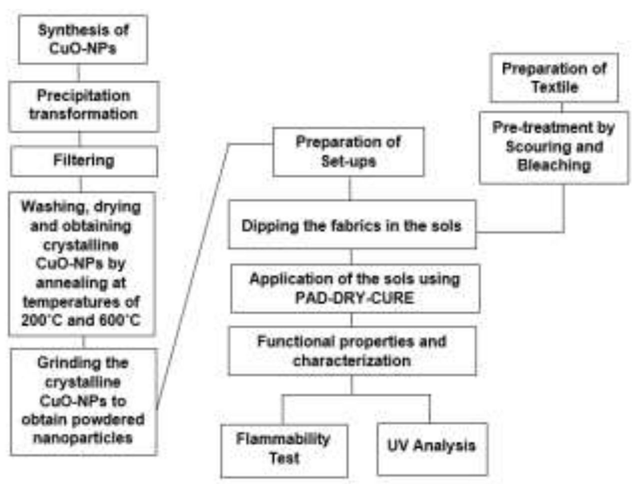


Figure 1. Research Paradigm

Synthesis of copper (II) oxide nanoparticles

This procedure was based adapted from the study of Luna et.al. (2015).

The CuO-NPs was synthesized through a standard procedure, through a chemical precipitation method. First, 8.5 g of copper (II) chloride dihydrate and 5.0 g of sodium hydroxide pellets were dissolved in a minimum amount of ethanol separately. To dissolve separately, the amount of ethanol was added and was as low as required. Through continuous stirring, sodium hydroxide solution was added dropwise to the copper (II) chloride dihydrate solution. This was done at room temperature. The solution's color changed during the reaction from green to cyan and black.

The black precipitate was copper hydroxide and was centrifuged. To remove the Na (salt) solution, the filtered precipitate was cleansed with ethanol and deionized water. Then, it was dried in the dryer with a temperature of 50°C. To finally crystallize CuO-NPs, the dried samples were annealed separately at temperatures 200 ° C and 600 ° C. Lastly, the annealed nanoparticles were ground to obtain powdered nanoparticles. The two nanoparticles have been labeled as CuO-NPs (200°C) and CuO-NPs (600°C) respectively. The reaction can be schematically described as: $CuCl_2$ + 2NaOH + 2NaCl.

Solution Preparation

This procedure was based adapted from the study of Przybylak et.al. (2015).

The mixture of tetraethyl orthosilicate (6mL) and non-ionic surfactant of ethylene glycol (2mL) produced silica hydrosol. Then, deionized water (6mL) was added to 180mL absolute ethanol and was stirred thoroughly using a magnetic stirrer for 15 min. Then, 4mL ammonia was added dropwise under the fume hood with constant stirring. Following this, 1g of CuO (bulk) was added into the tetraethyl orthosilicate while stirring for 15 min. Lastly, the solution was sonicated for 30 min using an ultrasonic irradiation device. This method was also done with CuO-NPs exposed at temperatures 200°C and 600°C respectively.

Pre-treatment, Pad-dry-cure, and Characterization

100% Cotton fabric was pre-treated by the scouring and bleaching method and was termed as Untreated Fabric. A homogeneous solution of CuO, CuO-NPs (200°C), and CuO-NPs (600°C) was prepared separately. The solutions were incorporated in the fabrics and used as a coating by a pad-dry-cure method using a padding mangle equipment and hot-air oven.

The dried cotton fabrics were dipped in different solutions, respectively. The fabric was put into the padding mangle equipment to obtain a uniform coating. Following this, a pressure of 4 kg cm2 was applied with a rotation speed of the cylinders of 5 rpm. The procedure was repeated thrice to enhance the attachment of the treatment onto the surface of the fabrics. Subsequently, the padded fabrics were air-dried and cured at 80oC for 5 mins in a hot-air oven. An exchange reaction between the –OH group of cotton and Si–OMe group of TEOS caused the nanoparticles to adhere to the fabrics. Hereafter, the fabrics were labeled as Untreated Fabric, CuO (bulk) treated Fabric, CuO-NPs (200°C) treated Fabric, and CuO-NPs (600°C) treated fabric, respectively.

Flame Retardancy and UV-Protective Tests

This procedure was based adapted from the study of Dhineshbabu et.al. (2014).

The flame-retardant properties of the samples were carried out by burning fabrics with a standard sample size of 7cm x 5cm in Trial 1 and 20 cm x 8 cm in Trial 2. It was conducted two times to obtain constant results. This was conducted at room temperature. The burning time of the fabrics was recorded by measuring the time when the fabrics were completely burnt. The UV-protective properties of the cotton fabrics were measured using Agilent Cary UV-Vis Spectrophotometer following the

AATCC Test Method 183-2004 which measures the Ultraviolet Protection Factor (UPF), Ultraviolet A-rays (UVA) Transmission, and Ultraviolet B-rays (UVB) Transmission to determine the UV-protection properties of textiles. Then, the results were categorized according to ASTM D6603 (Table 1) to determine the UV-resistance of the samples.

Table 1. UV-Protection Category according to ASTM D6603

UPF Range	Protection Category	Effective UVR Transmission, %	UPF Rating
15-24	Good Protection	6.7 to 4.2	15, 20
25-39	Very Good Protection	4.1 to 2.6	25, 30, 35
40 to 50, 50+	Excellent Protection	≤ 2.5	40, 45, 50, 50+

RESULTS

Flame Retardancy

The flame-retardant properties of different samples were observed by recording the burning time of the fabrics. It is shown in Table 2 that Untreated Fabric was burnt within 48 seconds for Trial 1 and 1 minute and 12 seconds for Trial 2. The second sample was the CuO (bulk) treated Fabric which was burnt within 1 minute and 6 seconds and 2 minutes and 4 seconds for Trial 1 and Trial 2 respectively. Followed this was the CuO-NPs (200^{0}C) treated Fabric which was burnt within 1 minute and 9 seconds for Trial 1 and 10 minutes and 9 seconds for Trial 2. Lastly is the CuO-NPs (600^{0}C) treated Fabric which was burnt within 1 minute and 33 seconds for Trial 1 and 10 minutes and 58 seconds for Trial 2.

Table 2. Burning Time of the Fabrics

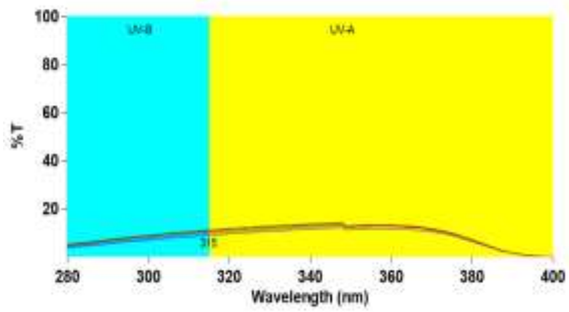
Type of Fabric	Trial 1 (7cm x 5cm)	Trial 2 (20 cm x 8 cm)
Untreated Fabric	48 sec	1min and 12 sec
CuO (bulk) treated Fabric	1min and 6 sec	2 min and 4 sec
CuO-NPs (200^{0}C) treated Fabric	1min and 9 sec	10 min and 9 sec
CuO-NPs (600^{0}C) treated Fabric	1min and 33 sec	10 min and 58 sec

UV Analysis

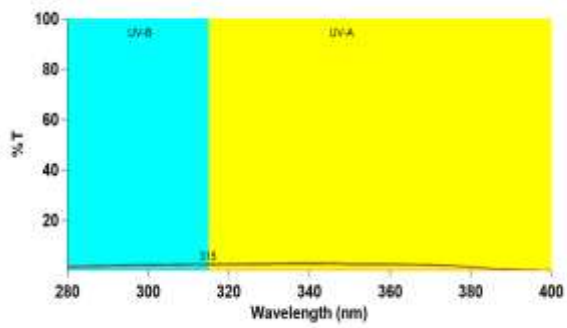
Table 3 shows the categorized UV analyses of fabrics that were carried out using Agilent Cary UV-Vis spectrophotometer following the AATCC Test Method 183-2004. The results were categorized according to ASTM D6603 (Table 1). The Untreated Fabric has 10.608 Mean Ultraviolet Protection Factor (UPF), 9.588 Mean Ultraviolet A-rays (UVA) Transmission and 7.193 Mean Ultraviolet B-rays (UVB) Transmission which was rated as Unclassified. The CuO (bulk) treated Fabric has 44.849 Mean UPF, 1.959 Mean UVA Transmission, and 2.096 Mean UVB Transmission which was rated as Excellent. The CuO-NPs (200^{0}C) treated Fabric has 86.186 Mean UPF, 1.150 Mean UVA Transmission, and 1.082 Mean UVB Transmission which was rated as Excellent. Lastly, the CuO-NPs (600^{0}C) treated Fabric has 107.054 Mean UPF, 0.892 Mean UVA Transmission, and 0.955 Mean UVB Transmission which was rated as Excellent.

Table 3. Burning Time of the Fabrics

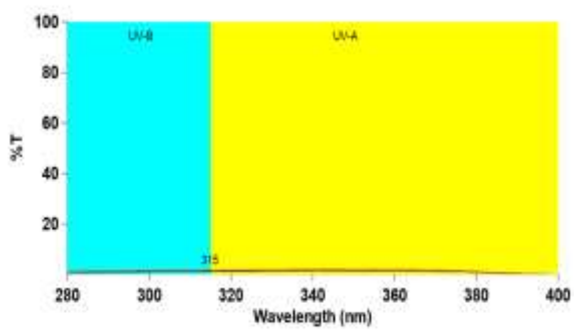
Fabrics	Mean UPF	Mean UVA Transmission	Mean UVB Transmission	Rating
Untreated Fabric	10.608	9.588	7.193	Unclassified
CuO (bulk) treated Fabric	44.849	1.959	2.096	Excellent
CuO-NPs (200^{0}C) treated Fabric	86.186	1.150	1.082	Excellent
CuO-NPs (600^{0}C) treated Fabric	107.054	0.892	0.955	Excellent



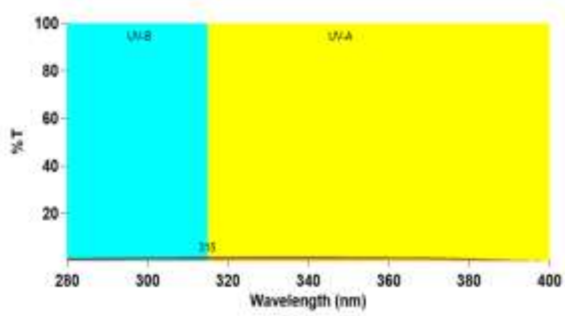
2a. Untreated Fabric



2b. CuO (Bulk) Treated Fabric



2c. CuO-NP's (200^{0}C) Treated Fabric



2d. CuO-NP's (600^{0}C) Treated Fabric

Figure 2. UVA and UVB Transmission Results

The figures above shows the UVA and UVB Transmission of different samples. As shown in Figure 2a, the Untreated Fabric has 9.588 Mean Ultraviolet A-rays (UVA) Transmission and 7.193 Mean Ultraviolet B-rays (UVB) Transmission. The CuO (bulk) treated Fabric (Figure 2b) has 1.959 Mean UVA Transmission and 2.096 Mean UVB. The CuO-NPs (200oC) treated Fabric (Figure 2c) have 1.150 Mean UVA Transmission and 1.082 Mean UVB Transmission. Lastly, the CuO-NPs (600oC) treated Fabric (Figure 2d) have 0.892 Mean UVA Transmission and 0.955 Mean UVB Transmission. The application of treatments inhibits the transmission of UV rays into the fabrics.

DISCUSSION

The contact of the fabrics with flame revealed significant differences in the time when they will be completely burnt, shown in Table 2. This showed the flame retardancy capacity of the untreated and treated fabrics. The Untreated Fabric was burnt the easiest and fastest as compared to treated fabrics. Treating the fabric with CuO (bulk) made its burning time longer since the application of metal oxide has something to do with the thermal degradation of the fabrics. Parallel to this, the addition of CuO nanoparticles to the fabric resulted in more flame-retardant capacity due to its size that improved the application of the treatment. However, the nanoparticles annealed at temperature 600^0C has more flame-retardant properties than the 200^0C nanoparticles. To fully support this claim, as stated by Rajendran et al. (2014), adding metal oxide nanoparticles such as copper (II) oxide on textile fabrics will improve its qualities including the fabric's flame retardancy. They concluded that by adding metal oxide nanoparticles on uncoated fabrics, the thermal stability and flame-retardant quality of the fabric will significantly enhance.

The classification of the results of the UV Protection based on the category of the UV protection according to ASTM D6603 is shown in Table 1. The UV analysis revealed significant differences between the UV protective properties of the fabrics shown in Table 3. The graph of the results shown in Figure 2 showed that the higher the amplitude, the higher its UVA and UVB Transmission and so, the lower its UV Protective Factor. The fabric that has the least amount of UPF is the Untreated Fabric. This fabric also has the highest transmission of UVA and UVB. However, the application of CuO (bulk) metal showed a significant effect on its UPF, UVA, and UVB Transmission as compared to the Untreated Fabric. To support this claim, according to the study of El-Nahhal et al. (2012), using CuO as a nanoparticle and applying it in the cotton it will have excellent protection against UV radiation. During the synthesis of nanoparticles, there were two samples. The first nanoparticle is annealed at temperature 200^0C and the second one was annealed at 600^0C. And based on the UV results, nanosizing the copper (II) oxide provided more UPF and less UVA and UVB Transmission. But the nanoparticles annealed at temperature 600^0C has more UV-protective properties than the 200^0C nanoparticles.

Based on the results from Flame Retardancy and UV Analyses, it was revealed that copper (II) oxide provides functional properties to the fabrics. The synthesis of nanoparticles, wherein the metal oxide was annealed at different temperatures for it to breakdown into smaller sizes produced more properties to the fabrics. The CuO-NPs (600^0C) treated fabrics contained the highest flame retardancy by having a long time of thermal degradation. It also had the least transmission of UVA and UVB which led to higher UPF.

Its functional properties were excellent because the higher the temperature the metal oxide was exposed, the smaller its size will become. And, the higher its attachment to the surface of the fabrics. Therefore, the copper (II) oxide was nanosized successfully and was the main reason for having higher functional properties than the CuO (bulk) treated Fabric. To support this claim, according to the study of Luna, Hilary, Chowdhury, Gafur, Khan, M., and Khan, R. (2015). It was noticed that with increasing the annealed temperature, the crystallization of the particles was increased because higher annealing temperature means higher energy given which results in more oxidation. Heating copper (II) hydroxide produced copper (II) oxide. In addition to this, according to Dhineshbabu et al. (2018), the use of metal oxide nanoparticles for the functionalization of cotton fabrics is effective since it was proven in his study that the nanoparticles firmly adhered to the surface of the fabrics. Multifunctional fabrics with nanomaterial treatments also display high durability and stabilized attachment against laundering. Nanoparticles also reduce the risk of toxicity because of the release of the nanoparticles.

CONCLUSION

The copper (II) oxide affects the flame retardant and UV-protective properties of cotton fabrics. Annealing the metal oxide with higher temperature break downs its size into a smaller size which increased the functional properties and attachment of CuO.

The copper (II) oxide nanoparticles were prepared by precipitation transformation, filtering, washing, drying, obtaining crystalline CuO-NPs by annealing at 200^0C, and 600^0C and grinding. CuO-NPs with tetraethyl orthosilicate cross-linker sols is obtained using the sol–gel method and then coated in the cotton fabric using the pad-dry-cure method. The nanoparticles were coated on the fabrics because of the chemical reaction between –OH group of cotton and Si–OMe group of TEOS. The fabric coated with copper (II) oxide (bulk) possesses good flame retardant and UV-protective properties.

On the other hand, the flame retardant and UV-protective properties of CuO-NPs treated fabrics were found to be better than that of the untreated and CuO (bulk) fabrics due to the size of the CuO nanomaterial. However, CuO-NPs (600oC) treated fabric has greater functional properties than the CuO-NPs (200oC) treated fabric because of the smaller size it has become during the synthesis of the nanoparticles. Therefore, the smaller the size of the nanoparticle, the higher will be its attachment to the surface of the fabrics and the higher its multifunctional properties will be.

Furthermore, the investigation described suggests that CuO-NPs (600^0C) cotton fabrics will be an effective multifunctional textile material for military applications and consumer use because of its high functional properties, low toxicity, affordability, and availability in the market. Gupta & Afshari (2018) also support this claim when they stated that the metal-based flame-retardants and UV-protective materials (metal oxide/salts and complexes) are low cost and considered to be environmental friendly (Gupta, B., & Afshari, M., 2018).

RECOMMENDATION

The following are then recommended for further improvement of the study. It is recommended to subject the fabrics to the washing durability test to determine the washing capacity of the coatings of nanocomposites. A tensile strength test is also recommended to determine if the fabrics have increased their tensile property. Lastly, cytotoxicity tests to know the effect of the treated fabrics to human cells.

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