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RESEARCH ARTICLE

Multifunctionalized cotton fabric using Cu nanoparticles

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Abstract

Cu nanoparticles colloidal solution was prepared by using cupper acetate in presence of an ecofriendly reducing agent [ascorbic acid] ,polyvinylpyrrolidone as protecting and stabilizing agent for nanoparticles and polyethylene glycol for protection of Cu nanoparticles against oxidation .the so prepared solution has been examined by UV/vis spectrometer and TEM .Cu nanoparticles solution was applied to cotton fabric in presence and absence of crosslinking agent butanetetracraboxlic acid BTCA. Incorporation of nanoparticles into fabric was detected and verified using SEM-EDX analysis as well as X- ray analysis. Functional Cu nanoparticle textile fabric offer great potential for a wide range of applications .Thermal stability of the treated fabric as well as antibacterial activities was improved. The treated fabric exhibits very high electrical conductivity .It is, therefore concluded that treatment of cotton fabric with nano-sized copper particles along with BTCA produces smart textiles fabric with multifunctional characteristics

INTRODUCTION

In recent years a shift to nanomaterialas a new tool to improve properties and gain multi-functionality is clearly seen. Nano - additives and nanocomposites have provided a fruitful area for polymer research, but the number of application to achieve a commercial reality is still very limited. (Barber et al 2009)[1]

The best nano material additive relies on both application and host polymer. Transition metal nanoparticles have attracted considerable interest from both medical and technological stand points, because they have a kind of property that plays a crucial role in applications (Cioffi et al 2005; Yoon et al 2006 and Ramyadevi et al 2012

Of the various metal nanoparticles, copper nanoparticles are popular by virtue of their potential use in a wide variety of fields such as optical, catalytic, electronic, and antifouling applications (Larsen and Noriega (2004); Wang et al (2004); Ryu(2011). Numerous methods have recently been developed for the synthesis of copper nanoparticles including chemical reduction (Biçer and Şişman(2010); Kumar et al (2012) ; Abdulla-Al-Mamun et al 2009; Guajardo-Pacheco2010; Yu et al 2009.) . Thermal reduction(Salavati and Davar 2009) radiation methods (Zhou et al ,2008) , microemulsion techniques (Lopez-Quintela , 2003) , laser ablation (Patel et al ,2005) polyol method (Park et al 2007) , and the DC arc discharge method (Charinpanitkul et al 2009). Among these methods, chemical reduction in an aqueous solution exhibits the greatest feasibility for further applications due to its simplicity and low cost (Brege et al 2009) .

The fundamentals of nanotechnology are a manifestation of the fact that properties of substrates undergo profound change when their size is reduced to the nanometer range Chattopadhyay and Patel 2010). Furthermore, a small amount of nanosize species can interfere with matrix polymer that is usually in similar size range, bringing up the performance of resultant system to an unprecedented level (Qian and Hinestroza, 2004).

The metal nanoparticles such as Cu are widely reported to have antibacterial activity (Ruparelia et al 2008). The bactericidal effect of metal nanoparticles has been attributed to their small size, and high surface to volume ratio, both allow them to interact closely with microbial membranes and it is not merely due to the release of metal ions in solutions (Morones et al 2005).

The textile industry is among other leading industries, which constitutes an area where nanotechnology is being implemented with full enthusiasm (Ward., (2003) . Nanotech-textiles are on the way of becoming the most popular textiles with their protective, functional and electronic features. It is particularly notable that one of most important advantageous features of nanotech-textiles is their protective properties (Hebeish et al (2014) Application of nanocopper colloidal solutions to cotton fabric not only improves its antimicrobial efficiency but also enhance the conductivity of cotton fabrics (Qufu., (2008)

In this study, the copper nanoparticles were synthesized as a reported method via chemical reduction method The prepared copper colloidal solution was applied to cotton fabric .Different properties acquired to cotton fabric were investigated.

2- Experimental.

2.1- Material.

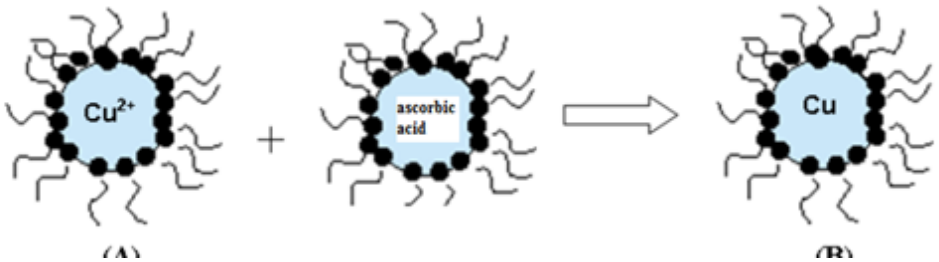
Fabric: mill-scoured and bleached cotton fabric were purchased from Misr Co. for Spinning and weaving, Mehala El kubra, Egypt

All of the chemicals used were of analytical grade and used as purchased without further purification. Copper [II] acetate hydrate salt of purity 98% [Merk] was dissolved in deionized water, polyvinylpyrrolidone, PVPK30, MW 40000, the PVP amount was kept at a molar ratio 5. Ascorbic acid $C_6H_8O_6$ was used as reducing agent, 1, 2, 3, 4 – butanetetracarboxylic acid as crosslinking agent

2.2- Synthesis of copper nanoparticles:

The typical synthesis procedure is illustrated in Scheme 1. First, the chelating agent [polyvinylpirrolidone, PVP K30, Mw 40000] was completely dissolved in 105mL of polyethylene glycol [PEG], and this solution was heated to the synthesis temperature under magnetic stirring. Once the synthesis temperature was reached, two PEG solutions, containing the proper quantities of the reducing agent [35 mL of $C_6H_8O_6$ solution] or the metal precursor [20 mL of $Cu[ac]_2H_2O$ solution],were added to the hot PVP solution; the reducing agent solution was poured first, followed by the metal ion solution a few minutes later. After Cu [ac] $_2$ additions, the green solution turned dark red, thus indicating the immediate nucleation of metallic copper particles. The solution was allowed to cool down to room temperature (Blosi, et al 2011)

The UV-vis absorption spectra of the colloids were recorded with a UV- vis spectrophotometer [Shimadzu corporation, UV – 1800].



2.3- Coating of Cotton Fabric Using BTCA/ Cu nanoparticles

An aqueous solution of 1, 2, 3, 4 – butanetetracarboxylic acid [30g/l] and sodium hypophosphite [NaH_2PO_2. H_2O] [6% W/W based on weight of [BTCA]. Mill scoured and bleached 100% cotton fabric were padded in the prepared solution in two dips and nips, and then squeezed to a wet pick – up of 100%. The padded fabrics were dried at 80C°for 5 min, then cured at 180C°for 3 min., The treated fabrics were padded in suspended solution of Cu nanoparticles and then squeezed to a wet pick – up of 100% and Finally dried at room temperature.

3- Characterization:

3.1- Transmission Electron Microscope [TEM]

The particle size of the prepared metallic copper was studied with a TEM [ZEISSEM10 – GERMANY] operating at 160 KV and at magnification power 50.000 Specimens for inspection by TEM were prepared by slowly evaporating one drop of prepared nanoparticle at room temperature.

3.2 UV–vis spectrophotometer

Many molecules absorb UV or visible light. In molecular absorbance spectroscopy a beam of UV or visible light is directed through samples. Some of the light may be transmitted through the sample. Light that was not transmitted through the sample was absorbed. An absorbance spectrum depicts what wavelengths of light are absorbed by a sample (Skoog 2007)

The UV/Vis absorbance spectrum, in this study, was obtained by passing different wavelengths of light ranging from 200 to 600 nm through the sample. The UV/Vis spectroscopy used was a Perkin Elmer lambda 900.

3.3- EDX analysis:

The energy – dispersive X – ray analysis [EDX] integrated with a phoenix energy – dispersive X –ray rector adds extraordinary capabilities to the entire system. It allows analyzing of elemental compositions down to boron including the light elements, such as carbon, nitrogen, and oxygen. The charging artifacts can be eliminated due to the existence of gas in the ESEM chamber (Yu et al 2005). In this study, the treated cotton fabrics were examined by the environmental scanning electron microscope [ESEM] at an accelerating voltage of 20 kv with accounting time of 100s.

3.4- Thermal measurements:

The stability and thermal behavior of treated and untreated cotton fabrics was measured by a thermo-gravimetric differential analyzer. The TGA scan was carried out using a computerized Perkin Elmer TGA series under a dynamic N_2 purging gas atmosphere at a constant rate of 50 cc/min and a heating rate of 50 C/min.

3.5- Antibacterial study of fabrics:

The antibacterial activities of the treated and untreated fabric were tested qualitatively by an inhibition zone method with E.coli as model bacteria. For qualitative measurement of antimicrobial activity, the samples were cut in small pieces, put together to form a circular zone and the antimicrobial activity was tested using modified agar diffusion assay [disc test]. The plates were examined for possible clear zone after incubation at 30C^{O}for 2 days. The presence of any clear zone around the fabric on the plates was recorded as an inhibition against microbial species.

3.6- Electrical Resistance:

The electrical conductivity of the dried metal – fabrics composite were determined at ambient room temperature [25^{0}C] using a Digital Multi-meter.

Electrical measurements were recorded by means of an electrical circuit composed by a Hewlett Packard 6634B System DC Power Supply and a digital Hewlett Packard 34401A Multimeter.

$$Conductivity = 1/R_S ;$$

Where Rs, Surface resistance was measured according to the American Association of Textile Chemists and Colorists Test Method 76-1995 (Lin, , et al. (2005)Two rectangular copper electrodes [20 X 30 mm^2] separated by 20 mm were placed on the fabric sample [30 X 60 mm^2] by a 1-kg mass. Surface resistance [R_s] is given by:

$$R_s(\Omega / square) = \frac{W}{D} R ;$$

Where R is the resistance measured by the multimeter, and W and D are the width of the sample and the distance between the two electrodes, respectively

4- Results and discussion:

4.1- Ultra – Violet / Visible spectroscopy:

Metal nanoparticles exhibit the absorption of visible electro-magnetic waves by the collective oscillation of conduction electrons at the surface (Takele et al 2006) This is known as the surface Plasmon resonance effect. The interest in this effect is the possibility of using it as a tracer for the presence of metal nanoparticles. In general, the nano sized copper particles typically exhibit a surface Plasmon peak at 556-580nm (Giorgioet al 2011) .The broadness of the absorption band probably arises from the wide size distribution of copper nanoparticles.

The exact position of the Plasmon absorption may depend on several factors including particle size, shape, solvent type and capping agent .As shown in Fig. 1 synthesized Cu nanoparticles exhibits maximum absorption at 570nm.This is rather the absorption band of copper particle or non – oxidized copper nanoparticles as reported.

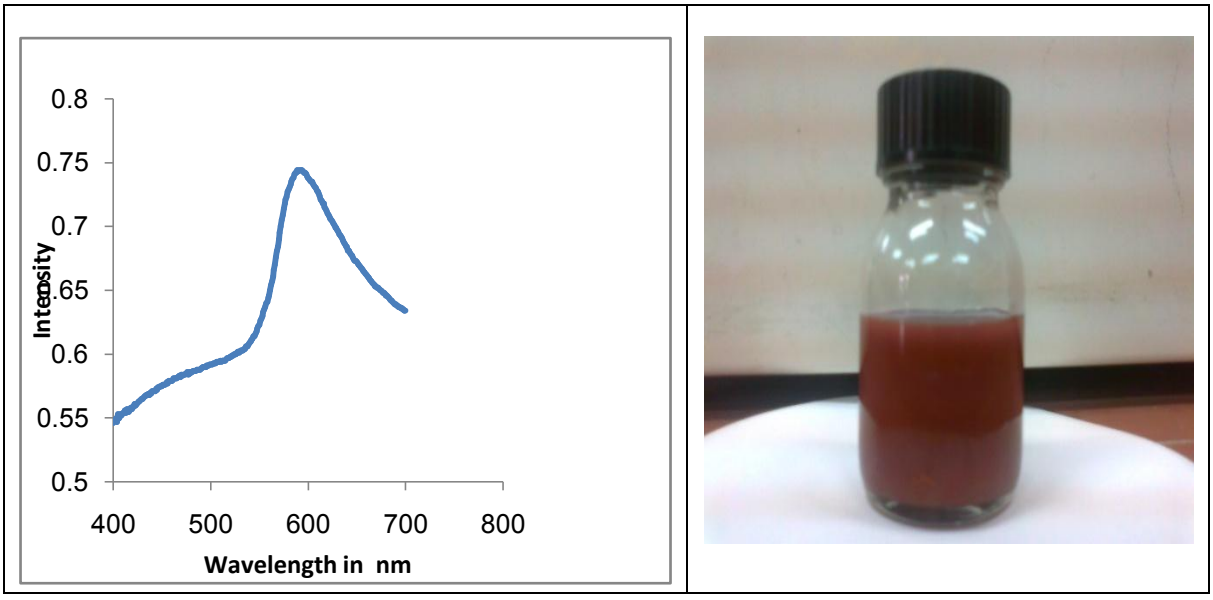


Fig. 1 a UV-vis absorbance spectra of Cu nanoparticles b] color of Cu nanoparticles suspension

4.2Cu nanoparticles mechanism

In our study PVP was used as a stabilizer for metal colloids, because of its availability, low cost and non toxicity. Once nuclei are formed, they tend to aggregate in order to decrease the total surface energy. This agglomeration, which can be a consequence of attractive van der waals forces between crystals, should be inhibited or limited to restrict the final particle size at the nanometric scale. One way to prevent nanoparticles agglomeration was the use of substances that lead to steric repulsion between individuals. PVP is an example of this type of growth and agglomeration inhibitors and improves the colloidal stability as it works both as size controller and polymeric capping agent because it hinders the nuclei from aggregation through the polar groups, which strongly absorb the copper particles on the surface with coordination bonds (Thi et al, 2011). The addition of ascorbic acid not only increases the reducing rate but also providing an antioxidant effect and improving the colloidal stability. This key role of the ascorbic acid addition can be explained by the fact that the precursor reduction rate is strictly linked to the following metal nanoparticle nucleation growth processes thereby leading to small particle dimensions and homogeneous particle size distribution.

The optical spectra of synthesized copper verify this hypothesis. As shown in Fig. 2 the characteristic peak appear at the same position at 570nm after 2 months, thus indicating that the PVP coating, together with the action of the antioxidant ascorbic acid as well as PEG, prevents the Cu – nanoparticles from oxidation, even if nano suspensions are exposed to air .

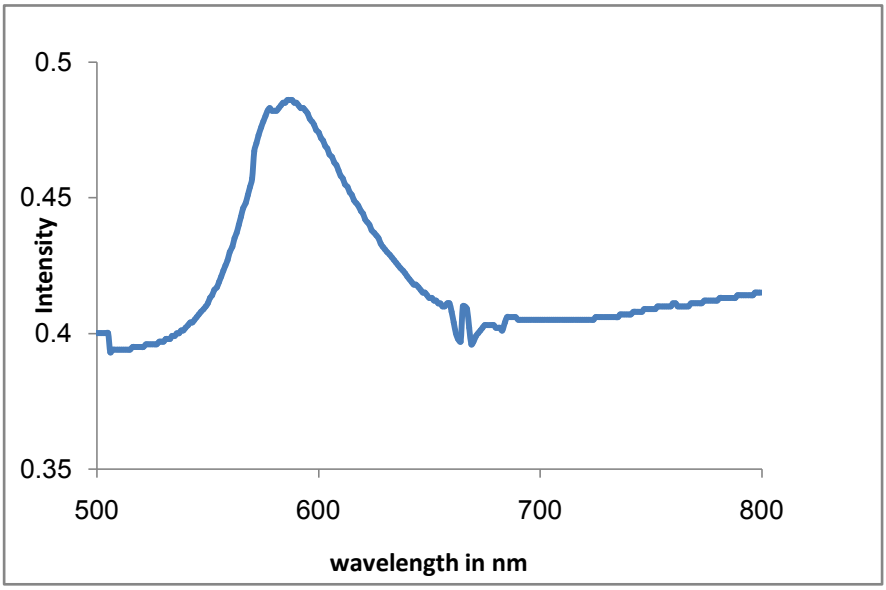
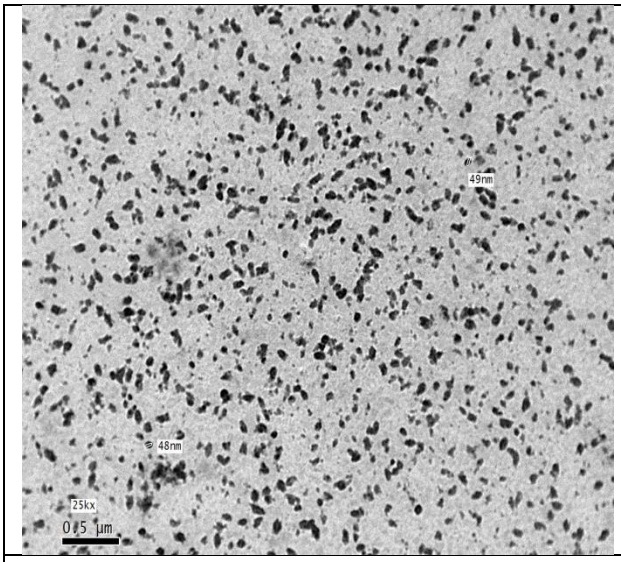


Fig. 2 UV-vis absorbance spectra of Cu nanoparticles colloidal suspension after two months

4.2 Transmission Electron Microscope [TEM].

The TEM images for the copper nanoparticles are shown in Fig3. Most of the particle average sizes are less than 100nm and the nanocrystals and triangles are also observed. The shape and size distribution illustrate that the size distribution of colloidal copper particles with a PEG to copper ratio used in this preparation [5: 1].Meanwhile a

size of Cu nanoparticles ranges between 29 and 50 nm. Considering these finding, we can say that the size distribution are homogenized.



29-50 nm

Fig. 3 TEM photographs of Cu nanoparticles colloidal suspension

4.3- SEM – EDX analysis of cotton fabric coated with cu nanoparticles.

A suitable way to gain information on copper nanoparticles incorporated into cotton fabric comes from SEM – EDX analysis. As shown in Fig. 4b Elemental X-ray mapping indicate the homogenous distribution of copper nanoparticles on cotton treated fabric, the deep pink color of copper nanoparticles also appear in Fig. 4a

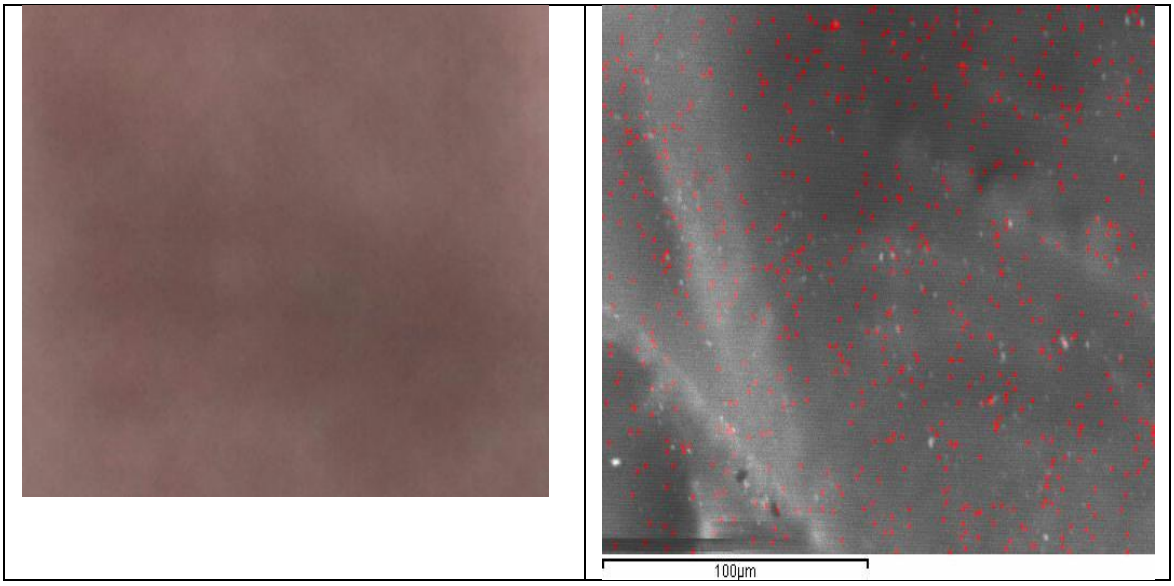


Fig. 4a It shows the color of Cu nanoparticles on cotton fabric;Fig. 4 b shows distribution of Cu nanoparticles on cotton fabric,

An elemental analysis of the particles was implemented by SEM equipped with an energy dispersive X- ray spectrum [EDX], which can provide a rapid quantitative analysis of the elemental composition. Fig.5 depicts the

quantitative analysis for cotton fabric treated with copper nanoparticles which confirms that the nanostructure contains about 36.847. Carbon, 24.66% oxygen, and 11.50% copper.

Fig.5 EDX spectra of cotton fabric treated with Cu nanoparticles

It can be exactly concluded that cu nanoparticles was present on the surface of cotton fabric. This may be because higher carboxylic acid groups of BTCA can maintain more particles of nano Cu

4.5- *X-Ray analysis*.

The X-ray diffraction pattern recorded for the copper nanoparticles is shown in fig 6. XRD peaks were using JCPDS files [JCPDS card on: 89-2838]. The XRD peak positions were consistent with metallic copper. As shown in Fig. 6 the sharp peak of the XRD pattern indicates the crystalline nature. The peaks at 43.3165, 50.44780^{o}corresponding to the miller indices [111], [200] represent face centered cubic structure of copper. The lattice constant of the unit cell is a = 3.615 A and its volume is 4.245 X 10^{-29} m^{-3}. The nanoparticles synthesized are found to be phase – Pure copper without any impurity phase such as CuO. Cu_2O and Cu $[OH]_2$.

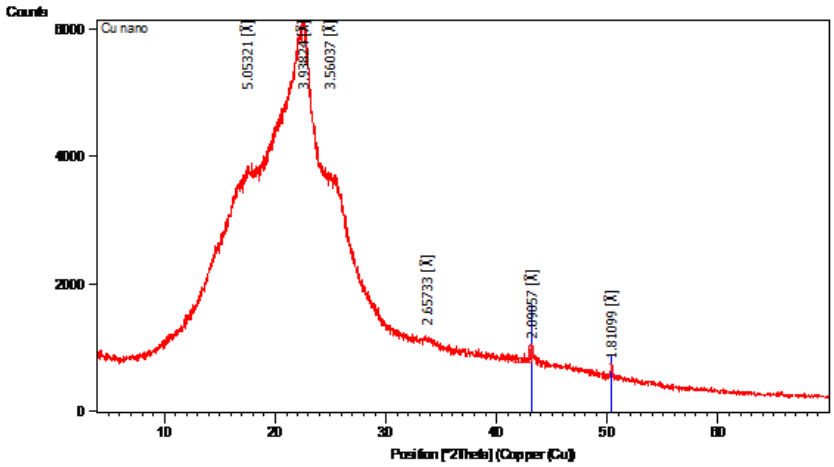


Fig. 6 XRD analysis for Cu nanoparticles on cotton fabric

4.6- Thermal analysis:

Thermo gravimetrical analysis [TGA] is widely used to investigate the thermal decomposition of untreated and treated cotton fabric to assess their relative thermal stabilities and the pathway of combustion and pyrolysis. TGA curves of treated and untreated samples (Fig.7a, 7b, 7c) as well as Table 1 revealed that the pyrolysis includes three mass loss stages: initial, main, and char decomposition steps as seen from [Fig. 7a]. The first stage [below 300C^{O}] shows change in physical properties and little mass loss, which is corresponding to the release of adsorbed water. Here, the damage to cellulose occurs mostly in the amorphous region. The main pyrolysis stage occurs in the temperature range of 300-370C^{o}at which the most of pyrolysis products are formed because the loss in mass is fast and significant. Referring to the literature, glucose is one of the main products, together with all kinds of flammable gaseous compounds. The TGA curve of the Cu nanoparticles treated fabrics[Fig. 7b,c] shows lower decomposition temperatures and mass loss, i.e. these stages happen below the thermal degradation of untreated cotton fabric which

started rapid thermal degradation at 300C^{o} and lost about 98% of mass. However, the treated cotton fabric [Fig. 7b,c]at the started degradation range of 200C^{o}and lost about 94% and 90% of mass in Cu, BTCA\Cu treated samples. Of particular interest is that the major mass loss for untreated fabric which occurred at 350C^{o}is about 75% whereas for the nanocopper treated cotton fabric without crosslinker is 64% and 59% for BTCA\Cu at 375C^{o}. Therefore it can be deduced that by the application of cu nanoparticles by different treatments enhance the thermal stability of cotton fabric as the formation of volatile pyrolysis products has been postponed .When cotton fabric is subjected to the thermal degradation the results revealed also that treatment of cotton fabric with BTCA prior to application of Cu-nanoparticle enhance not only easy care properties but also the thermal stability of cotton fabric.

sample	Evaporation		Decomposition temp.		Weight loss %
	Wt. %	Temp	T_0	T_∞	
cotton	4.51	100	307.7	350.16	**75,34**
cotton -Cu	5.15.	100	305	375	**63.7**
cotton –Cu -BTCA	5.53	100	310.33	375	**58.68**

Table 1: Thermal decomposition of cotton, cotton containing Cu nanoparticles and cotton –containing Cu nanoparticles along with BTCA

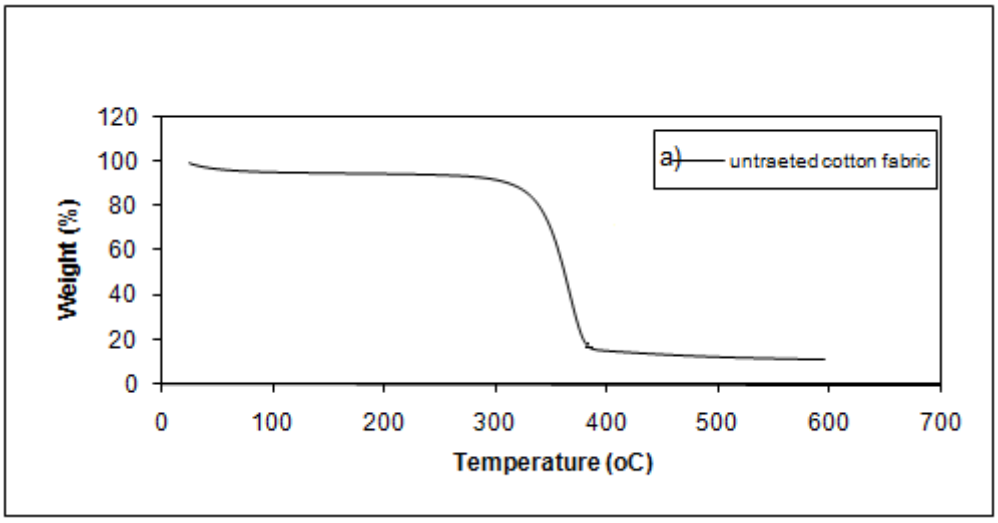


Fig. 7 a: TGA of cotton sample

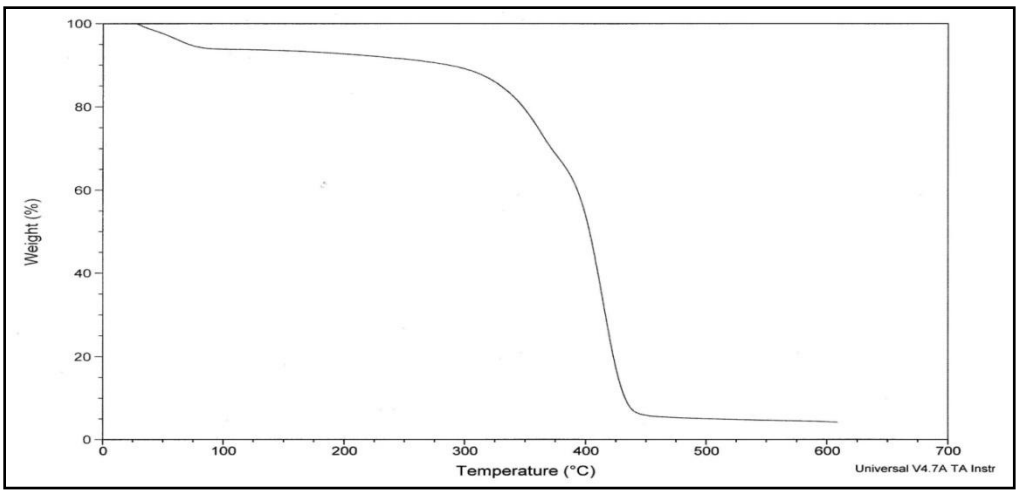


Fig. 7b: TGA of cotton fabric treated with Cu nanoparticles

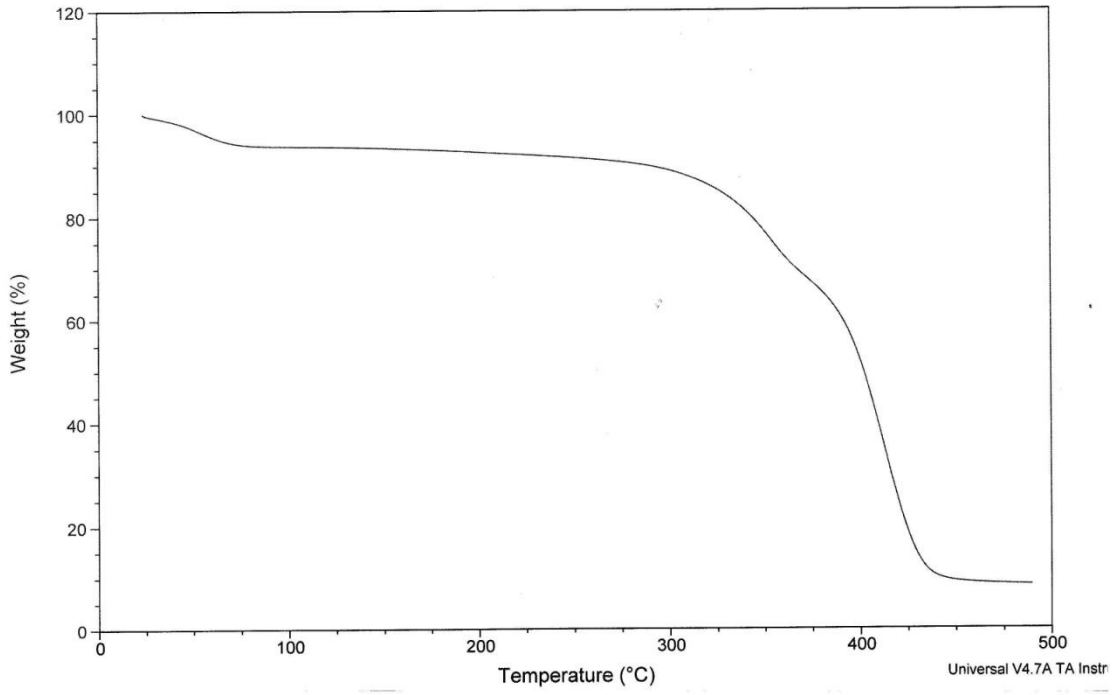


Fig.7c: TGA of cotton fabric treated with BTCA followed by Cu nanoparticles

4.7- Antibacterial activity:

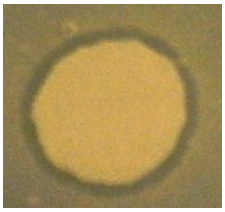
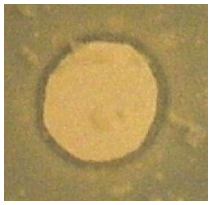
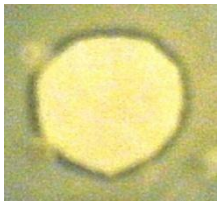
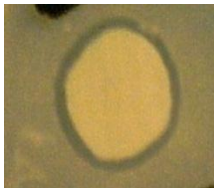
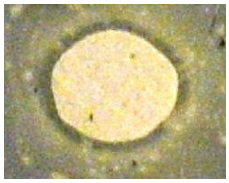
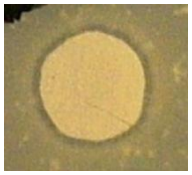
To investigate the antimicrobial activity of cu-nanoparticle treated cotton fabric, two fabrics were prepared, treated fabric without cross linking and the other one were treated with cross linker followed by treatment with copper nanoparticle.

The ability of Cu nanoparticle coated cotton fabric to inhibit bacterial growth on solid media was assessed using the inhibition zone test, which is commonly applied to wound dressing materials (Kumar.et al 2012) Table 2 clearly shows the antibacterial activity of the prepared samples. The bioassay was carried out using two gram – ve bacteria [*E. coli and P. aeruginosa*] and against two gram + ve bacteria [*St. aurous* and *B. Subtilis*] as per the disc plate technique.

The result of antibacterial activity of these fabric against *st.aureus(gm* +ve) and *E.coli* (gm –ve)have the highest sensitivity to Cu-nanoparticles and show increasing trend of diameter of inhibition zone in the sample prepared using BTCA crosslinking treatment.

Cu has the following mechanism to kill bacteria. It has been suggested that copper damages lipids, proteins, and nucleic acids via production of reactive hydroxyl radicals. In the presence of oxygen, many metals react with water to produce H_2O_2.which can damage cellular components via oxidation of copper. Cu can also directly damage cellular components. (Jinyu Li & John J. Dennehy (2011) The treatment of cotton fabric with BTCA prior to treatment with Cu nanoparticles retain or keep the antibacterial activity of Cu nanoparticles treated cotton fabric , beside the favorite functionality acquired to cotton fabric by using BTCA.

Table 2 the antibacterial activity of treated cotton fabric

Sample	Inhibition zone [mm] / 1cm sample			
	[G +ve]		[G -ve]	
	St. aureus	***B. subtilis***	***P. aeruginosa***	***E. Coli***
Cu	16 	14 	14 	14 
Cu\BTCA	16 	14 	13 	14 

4.8Electerical conductivity measurement:

The results of the electrical conductivity test for the materials under investigation are given in table 3. Obviously the untreated cotton fabric has a very high surface resistance i.e. very low conductive surface per unit length [5.502 x 10^{-10}] but the copper treated cotton fabric reduces the surface resistance significantly as shown in table3, the highest conductive surface [3.9 X 10^{-6}]. The cotton fabric which was treated without BTCA treatment shows low conductive surfaces [3.74X 10^{-6}] which demonstrates that the cotton fabric previously treated with BTCA enhanced the surface conductivity this may be due to that BTCA lead to deposition of more copper on the fabric?
.

Table 3: the electrical conductivity test

Sample	Conductivityσ [Conductivity] $\Omega^{-1}\cdot m^{-1}$
Cu	3.74X 10^{-6}
Cu\BTCA	3.9X10^{-6}

Blank	$5.502x10^{-10}$

5. Conclusion

Cu nanoparticles solution was prepared successfully using cupper acetate in presence of an ecofriendly reducing agent [ascorbic acid] ,polyvinylpirrolidone as protecting and stabilizing agent for nanoparticles and polyethylene glycol . The surface Plasmon band of the UV–vis spectrum at 570 nm indicates the existence of copper nanoparticles .The prepared solution was stable for more than two months. Cu nano

Particles solution was applied to cotton fabric in presence and absence of crosslinking agent butane-tetra carboxylic acid BTCA. Incorporation of Cu nanoparticles into fabric was detected SEM-EDX analysis as well as X-ray analysis. The copper nanoparticles treated fabric displayed antibacterial activity toward the tested pathogenic strains of two gram – ve bacteria [*E. coli and P. aeruginosa*] and against two gram + ve bacteria [*St. aureus* and *B. Subtilis*]. *St.aureusgm* and *E.coli* have the highest sensitivity to Cu-nanoparticles treated fabric. The treated fabric are more thermal stability than the untreated one .The electrical conductivity of the treated Cu nanoparticle cotton fabric totally improved. Also we can conclude that treatment of cotton fabric by BTCA as a crosslinker not only enhance easy care properties but also the thermal stability of the treated fabric in addition such pretreatment not affect on the electrical conductivity or the antibacterial properties of Cu nanoparticle treated fabric .the functional Cu nanoparticle treated fabric have a great potential for wide range of applications. i.e. medical as well as conductive textile.

Acknowledgement

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