



Facile route for synthesis of flame retardant, antibacterial and conductive fabrics

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Abstract

Textile-based materials constitute the majority of market products; however, their uses are limited in some applications due to their low thermal stability and high flammability hazards. Therefore, in this study, we developed a conductive cotton textile fabric based on a green fabrication method using different composites of three different dimensional nanomaterials: cellulose nanocrystals, graphene, and polyaniline. The developed smart-coated cotton samples showed good flame-retardant behavior, good antibacterial properties, promising ultraviolet protection and improving thermal stability. The treated cotton fabric achieved the highest flame retardancy recording a zero burning rate compared to 115 mm/min for blank cotton fabric. Additionally, the antibacterial activity of the treated fabric was significantly improved, achieving a clear inhibition zone of 35 mm compared to zero for untreated cotton. Furthermore, the developed coated textile material records high electrical conductivity. This study affords green guidance for design and fabrication of green multifunctional coatings useful for various avenues and applications.

Keywords: Cellulose nanocrystals, Textile fabrics, Conducting polymers, Flame retardancy, antibacterial property.

1 Introduction

Natural and synthetic fibers, and their blends, are used in numerous applications for textile materials [1]. However, these textiles' characteristics should align with the necessary functions [2-5]. Thus, some functions should be incorporated to achieve the demand properties. However, their application could have been by their cellulose-based precursor molecule's poor thermal stability and high flammability [6]. For this reason, it is imperative to treat the surface of textile fabrics with an antibacterial agent [7]. There are numerous compounds have been used as flame retardants to preserve cotton fabric against fire hazards. Different flame-retardant materials, such as halogenated materials nitrogen and phosphorus-based compounds, have been utilized [6-9]. Recent advancements in flame retardants have been made by the use of nanomaterials alone or through surface engineering using organic or inorganic phosphate-based materials such as clay layers and carbon nanotubes, metal hydroxide nanoparticles, and

inorganic nanotubes [10-11]. Numerous nanomaterials have been employed to make textile fibers flame-resistant stemming from the synergistic effect of cooling and the formation of reinforced char barrier [12]. Adding multiple functions to textile materials to impart flame retardancy, electrical, UV protection, mechanical, and antibacterial behaviour has recently attracted the attention of many researchers [13-14]. On the other hand, nanocellulose and cellulose nanocrystals (CNC) offer numerous benefits over natural cellulose, including high strength, transparency, high surface area, low density, biodegradability, and strong mechanical qualities [15-18]. Nanocellulose is used in various applications as a component of nanocomposite materials to enhance the mechanical, thermal, and ionic conductivity properties of polymers [19-20]. The most popular method for obtaining CNCs is the strong acid hydrolysis process. The two chemicals with the highest crystallinity indices were H_2SO_4 and H_3PO_4 [18]. Phosphoric acid has shown its potential to be a promising substitute for sulfuric acid for the hydrolysis of cellulose due to its non-corrosive, non-oxidizing, non-toxic, and inexpensive properties [21-24]. To create molecules that are biocompatible or flame-resistant, phosphorylation is seen to be a promising environmentally friendly substitute for these changes. On the surface of cellulose, phosphoric acid hydrolysis results in the production of anionic groups. To obtain substances that are biocompatible or flame-resistant [25].

Interestingly graphene sheet (GRP) is a highly fascinating material for research and applications [26-27]. Additionally, the capability of graphene sheets to produce compact char layers, which function as a physical barrier for flammable degradation products and slow down the degradation process of polymer, results in the new polymer nanocomposites having additional flame retardancy qualities [28-29]. Recently, significantly less complicated methods for producing graphene have been demonstrated that are also less expensive and risky. Porous carbon compounds generated from the recycling of waste agricultural products provide cost-effective and green precursors for nanoporous carbon sheets for diverse applications [30]. Differently nanostructured electrically conducting polymers have highly intriguing physical and chemical characteristics, making them excellent for various uses [31-35]. Polyaniline (PANI) is a conducting polymer widely studied and demanded for many applications [36]. PANI may serve as a flame retardant by strengthening the char during the condensate phase [37-38]. Overall, the synthetic availability of polyaniline, their adaptation to various application procedures, lack of skin irritancy, and high antimicrobial activity offer opportunities for their use in the development of antibacterial textiles [39]. The conjugated polymer polyaniline has a biocidal effect because it was synthesized with positive charges on its backbone chain, [40]. This approach has been studied the synthesis and treatment of three different nanomaterials with different dimensions as effective flame retardant and antibacterial agents to cotton textiles. The nanocomposites prepared comprised cellulose nanocrystal phosphate (P-CNC), conducting polymer (polyaniline) and graphene. The treated textile fabrics' electrical, thermal, mechanical and antibacterial properties were studied and optimized.

2. Experimental section

2.1. Material

Commercial Cotton Textile Fabric©, Hydrochloric Acid (HCl) was supplied from Merck-85% Phosphoric Acid was supplied from SDF- Sulphuric Acid 98% was supplied from ALPHA-

Sodium Hydroxide was supplied from SDF –Ammonium Persulfate was supplied from SDF- Aniline was supplied from SDF- Cotton Waste Clothes –Mandarin Peels –Deionized Water (DI).

2.2. Preparation of Polyaniline nanofibers PANI-NFs

In a glass beaker containing 1 molar hydrochloric acid solution, disperse 0.91 ml of distilled aniline, then magnetic stirring; This is followed by dissolving 4.25 g of APS in hydrochloric acid solution in the second beaker. Then, dropwise the APS solution in the first beaker, followed by magnetic stirring for 3 hr. The solution was filtrated and washed with DI to remove excess aniline and acid to get a wet green polymer.

2.3. Synthesis of Graphene (GR)

Mandarin fruits were washed with DI water; the peels were removed and dried in the sun for seven days, then cut into small pieces and dried for 24 hr at 80 °C. Then, they were ground and dried at 80 °C under vacuum for 20 h. Then, the powder samples were carbonized in a muffle furnace at 800°C with a heating rate of 5 °C /min⁻¹ and a holding time of 1 hr. Then, the samples were washed with DI water, filtered, and dried. Then, the obtained black solid particles were ground using ball milling for 6 hr. The sample was washed and, the nanoporous carbon materials were separated from the liquid by centrifugation at 3600 rpm for 15 min. The supernatant was decanted, and the precipitate was obtained and dried to get powder.

2.4.1. CNC extraction process

10 g of the cotton waste clothes was cut into small pieces and washed with a bleaching agent (1M) of sodium hydroxide for 1 hr to remove the impurities and most non-cellulosic substances. The process is followed by washing with distilled water it reaches a neutral pH. This is followed by drying. Then, the obtained absorbent cotton microfibers (CMF) were hydrolyzed with 50 wt% H₂SO₄ solution at 40 °C for 30 min. Cold DI was added to terminate the reaction. The mixture was centrifuged thrice at 4000 rpm for 15 min to remove excess acid. The milky white CNCs suspension was sonicated for 10 minutes to homogenize the generated nanocrystalline cellulose.

2.4.2 Preparation of phosphorylated-modified CNCs

P-CNC was prepared by phosphorylation of CNCs (0.5gm) using 85% phosphoric acid followed by stirring at 180 °C for 3 hr in a necked flask equipped with a condenser, cooling to room temperature in an ice bath, washing by centrifugation five times and denoted as (P-CNC1).

2.5. Preparation of smart coating suspensions

2.5.1. Preparation of GRP+PANI-NF

In one beaker containing 50 ml of DI, 1gm of GR and 0.5 gm of PANI-NF were dissolved, followed by ultrasonication for 10 min to prepare homogenous suspensions denoted as GRP-PANI-NF.

2.5.2. Preparation of P-CNC1-GRP-PANI-NF

In one beaker containing P-CNC 1 (0.5gm) ,0.5gm of PANI-NF and 1gm of GR were dissolved, followed by ultrasonication for 10 min to prepare homogenous suspensions denoted as P-CNC1-GR-P-PANI-NF.

2.6.1. Application of smart coatings on textile fabrics

The cotton samples were cut in 20 cm × 25 cm and were immersed in each coating dispersion prepared individually for 10 min. Then, samples were removed and squeezed out, repeated three times. Afterward, the samples were dried in the air, followed by a curing step in the oven **at 120 °C for 5 min** . The obtained modified fabrics were denoted as C-GRP+PANI-NF, C-P-CNC1-GRP-PANI-NF; this is in addition to blank cotton C as shown in table 1.

Table 1. The compositions and abbreviations of treated textile fabrics

Sample code	P-CNC1 (wt.%)	PANI-NF (wt.%)	GRP (wt.%)
C	0	0	0
C-GRP-PANI-NF	0	25	75
C-P-CNC1-GRP-PANI-NF	25	25	50

2.7. Characterization

The surface and dimension of CNC and P-CNC1 were evaluated using a Transmission electron microscope (TEM) using JEOL (JEM-1400 TEM). The XRD analyses of graphite and graphene samples were performed using a Rigaku X-ray diffractometer (Ultima IV) running at 40 kV and 30 mA with a range of 3–41 2θ angle. Thermo gravimetric analysis was conducted using DTG 60 from room temperature to 750 °C at a heating rate of 10 °C/min under nitrogen atmosphere flow. The flammability properties of untreated and treated textile fabrics were studied using a Fire Testing Technology UL94 flame chamber based on modified ISO 3795 [1,8,41-42]. The antibacterial activity was investigated using the AATCC standard test method 147–2004. The mechanical properties were evaluated using tensile testing machine model H1-5KT/S.

3. Results and discussion

3.1.1. Green synthesis tool of textile fabrics coatings

The cotton fabric properties were greatly improved using green, smart and innovative nanocoatings. The nanocoating was fabricated from P-CNCs prepared from cotton wastes via acid hydrolysis, GRP sheets prepared from fruit byproducts (mandarin peels) by carbonization followed by pall-milling and PANI-NF prepared using oxidizing agents. The obtained green coating was the coated-on cotton fabrics as indicated in Table 1 and schematically presented

in Fig.1. The properties of CNCs prepared were evaluated using microscopic (TEM) and spectroscopic (IR) tools, however, GRP sheets were characterized using XRD.

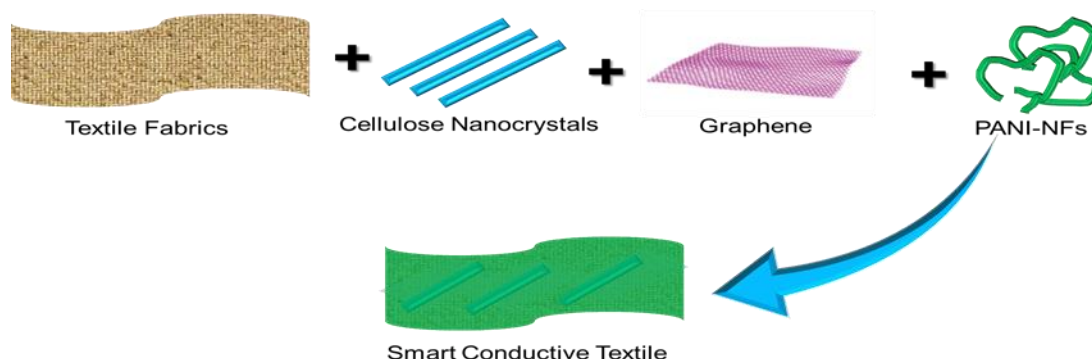


Fig. 1. Schematic diagram representing the green synthesis of renewable textile fabrics coatings and their application on textile fabrics.

Fig. 2 represents the TEM image of CNCs which reflects the existence of CNCs rods in a nanoscale with dimension of 10 nm (Fig. 2a), however, upon phosphorylation the CNCs size was increased to 18 nm (Fig. 2b) .

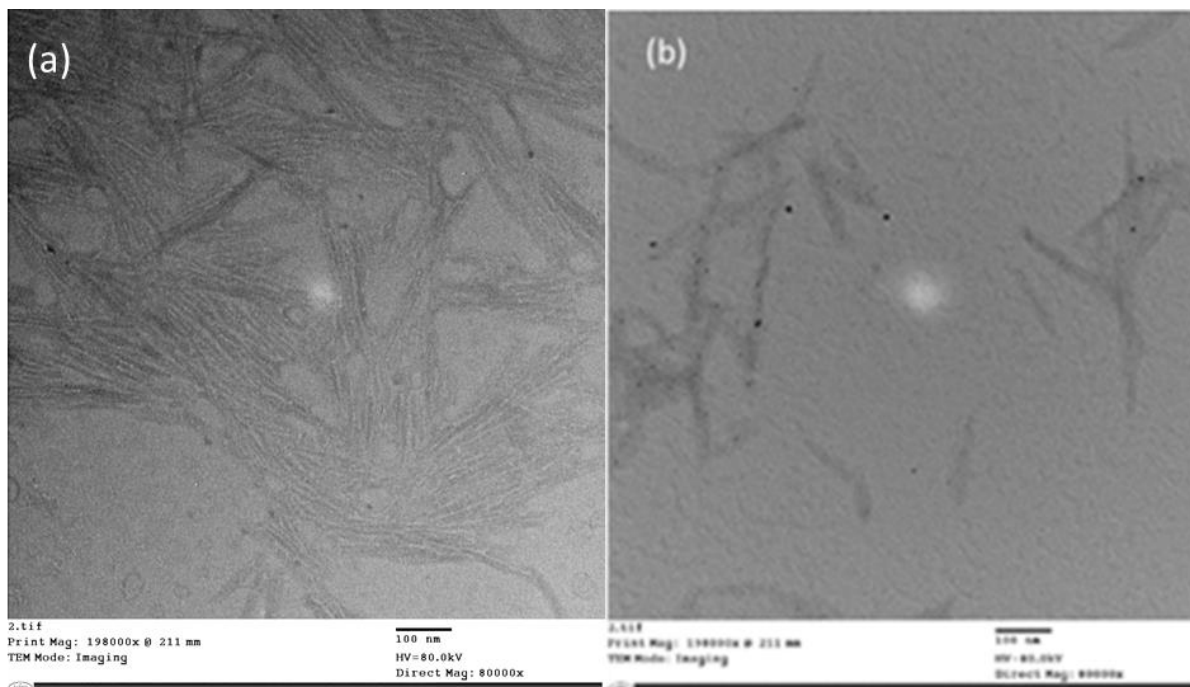


Fig. 2. TEM images of (a) CNC and (b) P-CNC1.

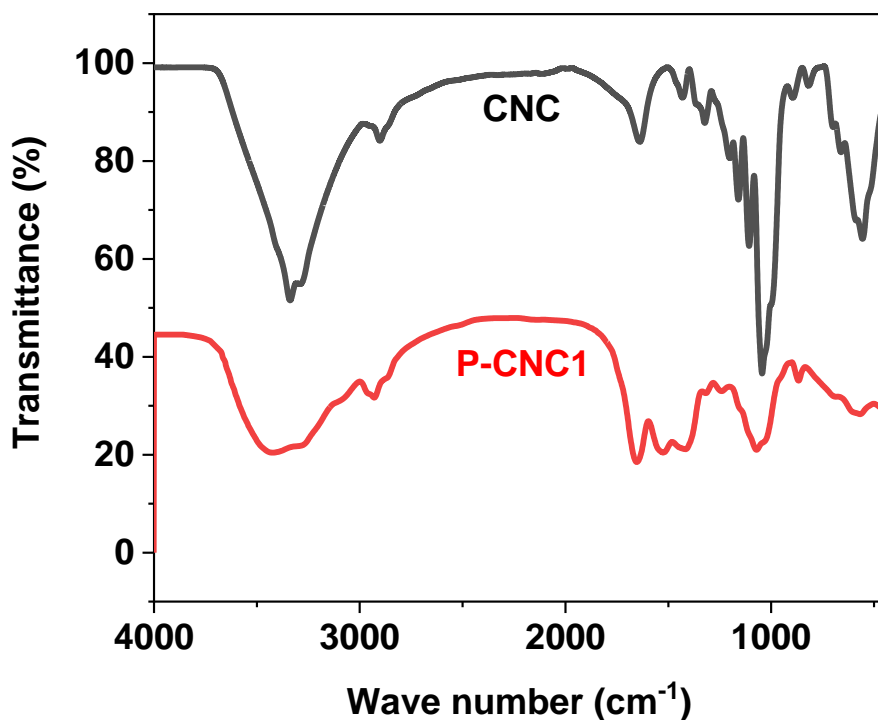


Fig. 3. FT-IR spectra of CNC and P-CNC1.

Fig. 3. represent the characteristic absorption peaks of CNC and P-CNC. Thus, the absorption peak detected at 1640 cm^{-1} corresponds to deformation vibration of adsorbed water of CNCs [43-44] , however, the strong broad band observed at 3330 cm^{-1} corresponds to stretching vibration of O-H [45-47]. Moreover, the absorption peaks positioned at 1045 and 1160 cm^{-1} are ascribed to C-O-C glycoside bond of β -1,4-glycosidic bond in cellulose nanocrystals and bending vibration of C-C, respectively [43-44]. Furthermore, the absorption peaks noted at 2904 and 899 cm^{-1} are attributed to asymmetric stretching vibration of C-H and stretching vibration of C-H, respectively[48]. While, the absorption peaks situated at 1430 and 1320 cm^{-1} are ascribed to symmetric bending vibration of CH_2 and bending vibration of C-H, respectively [45-46]. For P-CNC1 new absorption peak is detected at 1235 cm^{-1} which is ascribed to stretching vibration of P=O [49-50]. For GRP sheet exfoliated from graphite based mandarin shell XRD was used to affirm the synthesis process. The XRD pattern of Graphite attained upon carbonization of mandarin shell (M-GRP0) which indicated the sharp 002 peak situated at 26.5° as found in commercial graphite which is attributed to interlayer spacing of stacked graphite layers, this is in addition to other peaks which elucidated in our previous report. [46]. On the other hand, the thermal stability of untreated and treated cotton fabrics was evaluated using thermogravimetric analysis and data tabulated in Table 2. Fig. 4 represents the thermogram of blank textile which revealing three degradation steps as illustrated by TGA curves. The first step was detected at a temperature below 100°C and can be attributed to moisture in sample While the second peak was found to have occurred with the maximum temperature of 280°C relating to the degradation of functional groups of cellulosic fiber. The last degradation step

seen at 371 °C is for decaying the remaining carbon structure leaving a char yield of 4% at 750 °C [46] . In the case of samples treated with polyaniline and graphene , the decomposition of PANI chains on the surface of textile fabrics, first forming char layer rich with nitrogen species that protect the textile fabric from excessive thermal decompositions [33]. Additionally, the ability of graphene sheets to produce compact char layers, which function as a physical barrier for flammable degradation products and so slow down the degradation process of textile by delaying the mass and heat transfer process while the uncoated material was burned completely with higher mass loss which is also appearing from thermal analysis results. This enhances the char residue of developed textile samples at 750 °C from 4.4 to 16.7 % as indicated in Table 3 and Fig.4. The treated samples with P-CNC show initial mass loss below 100 °C which is assigned to trapped moisture in coated samples. However, the first decomposition step temperature which corresponds to the decomposition of treated cotton was reduced, this is due to the decomposition of phosphates that exist in the coating layer and the release of phosphoric acid which catalyze the dehydration of cellulose of cotton forming char barrier which protects textile from further degradation and prevent further mass loss. So, char residue increases for C-P-CNC1-GRP-PANI-NF to 33.5%. This indicated the improved thermal stability of developed textile fabrics compared to untreated one. Furthermore, the maximum degradation temperature (Tmax) of all samples was determined from DTG curves and their results were summarized in Table 3.

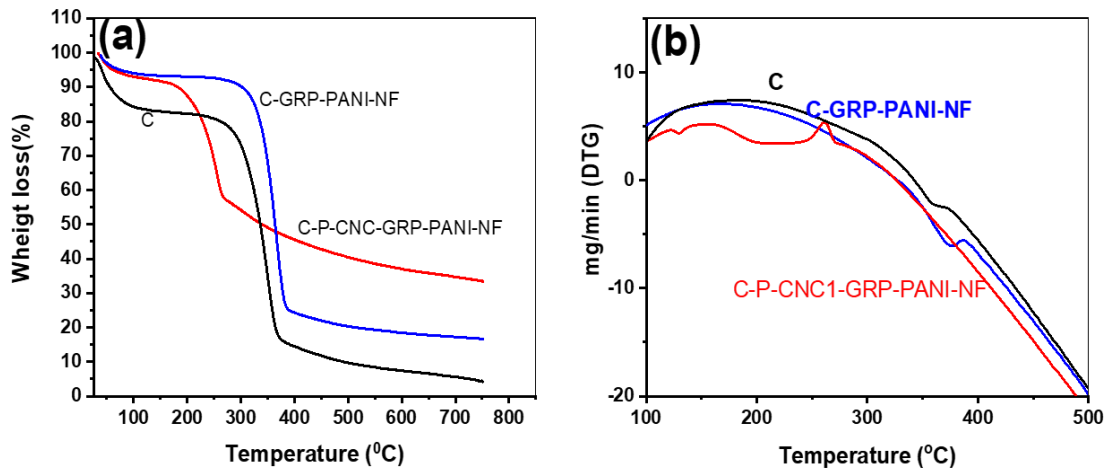


Fig.4. TGA and DTG thermograms of uncoated and coated cotton fabrics

Table 2. Thermogravimetric data of uncoated and coated cotton fabrics

Sample code	T _{onset} (°C)	Maximum weight loss (°C)	Char Residue (wt.%)
C	280	368	4.4
C-GRP-PANI-NF	298	375	16.7
C-P-CNC1-GRP-PANI-NF	183	201	33.5

3.2. Flammability properties

The flammability properties of untreated, treated cotton textile fabrics, untreated C and treated samples were evaluated according to modified ISO 3795. The rate of burning of different textile fabrics was tabulated in Table 3. The rate of burning of blank C was found to be 115 mm/min, respectively. which burnt very fast once ignited. In C-GRP-PANI-NF composites, the rate of burning was recorded as 77.24 mm/min which is reduced compared to C blank. This was due to the introduction of PANI-NF and graphene structure into the textile which improve the fire retardancy [51] . After burning, the coated material retains the original fibrillar morphology of cellulose, the resulting structure prevents the formation of gaseous carbon oxides by restricting the access of oxygen to cellulose and also, the porous structure of graphene offered significant restriction for the emission of toxic gases upon combustion . while the uncoated material was burned completely with higher mass loss which is also appearing from thermal analysis results[52]. With the addition of P-CNC1 to the treated samples as C-P-CNC1-GRP-PANI-NF the rate of burning was also reduced to zero. This was because Phosphorylated cellulose nanocrystals (P-CNC) achieved the formation of char layer upon the release of phosphoric acid which catalyze dehydration reaction of cellulose material leading to char .The rate of burning of These samples was zero due to the synergetic effect of all composites added. The positive fire retardancy behavior was due to the good char barrier formed due to combined effect of phosphorus compounds and graphene sheets which strengthen the physical barrier created based on graphene layers, hence, prevents the escape of degradation combustible gases to flaming zone[26,46] . The strong protective char achieved zero rate of burning as reflected in Table6 (high-class flame-retardant textile) [7,33,46].

Table 3. The flammability properties of untreated and treated textile

Sample code	Burned distance (mm)	Time of burning (sec)	Rate of burning(mm/min)
C	112	58.27	115
C-GRP-PANI-NF	112	87	77.24
C-P-CNC1-GRP-PANI-NF	112	0	0

3.4. Electrical conductivity and antibacterial properties

The electrical conductivity of coated and uncoated textile fabrics was assessed based on electrical resistivity (Table 4). The electrical resistance of uncoated blank C was more than 200 MΩ (which evaluates the inefficiency of material to hold energy or behave as an insulating material); with the incorporation of conducting polymer (PANI-NF) and graphene on fabric, the electrical conductivity increased, recording electrical resistivity of 12 KΩ. The electrical resistance to the motion of electrons decrease compared to the untreated fabric; This was due

to the π -conjugating system of PANI-NF. This higher electrical conductivity is ascribed to the intrinsic electrical conductivity of GRP, which acts synergistically with the electrical conductivity of one-dimensional PANI, affording good electrical conductivity fabrics suitable for various high-tech applications [23,33,53]. However, with the incorporation of P-CNC1, the electrical resistance increases but is still better than the blank untreated fabric. This may be due to the steric hindrance to the motion of electrons brought with the incorporation of both P-CNC1 and PANI-NF. This could be due to the rich phosphate groups attached to P-CNC1, which afford superior electrical insulation via localization of hopping electrons over PANI-NF.

Table 4. Electrical resistance of treated and untreated fabrics

Sample Code	electrical resistance
C	More than 200 M Ω
C-GR+PANI	12 K Ω
C-P-CNC+GR+PANI	100 K Ω

Antimicrobial textiles can contribute to the fight against antibiotic-resistant pathogenic microorganisms. The antibacterial properties of the treated and blank untreated textile fabrics have been tested against *staphylococcus aureus bacteria* and *E-coli bacteria*. The untreated textile fabrics C exhibited no antibacterial effect and has a negative effect toward the inhibition of bacterial growth as the clear inhibition zone for C was recorded as zero, as indicated in Fig. 5. Textile fabric samples coated with PANI chains and graphene have no effect against *E-coli bacteria (-ve)*, as The antibacterial inhibition zone for C-GRP-PANI-NF was found to be zero mm against *E-coli bacteria(-ve)* and zero mm against *staphylococcus aureus bacteria (+ve)*. The main reason for this antimicrobial effect is the chains of polyaniline in a doped state possess a significant positive charge and are capable of strong electrostatic binding with cell membranes of gram-negative bacteria and this plays a significant role in the manifestation of the antibacterial activity [40,54]. Samples coated with P-CNC have an antibacterial effect as, the clear inhibition zones for C-P-CNC1-GRP-PANI-NF was (35 mm) against *staphylococcus aureus bacteria (+ve)*. The explanation for this is that (P-CNC) can cause a change in the cell wall structure and the disturbance of the selective permeability of the cytoplasmic membrane. This destruction may lead to osmoregulation damage resulting in a decrease of metabolic functions [46,55,56]. The inhibition zone against *staphylococcus aureus bacteria (+ve)* was greater than that for *E-coli bacteria (-ve)*. This may indicate that gram-positive bacteria are more sensitive to the phosphate presence than gram-negative.

Table 5 . Inhibition zone of untreated and treated fabrics against *E-Coli and Staph-cocci*

Sample	Inhibition zone against (+ve) bacteria	Inhibition zone against (-ve) bacteria
C	0	0
C-P-CNC1-GRP-PANI-NF	35	10
GRP-PANI-NF	0	0

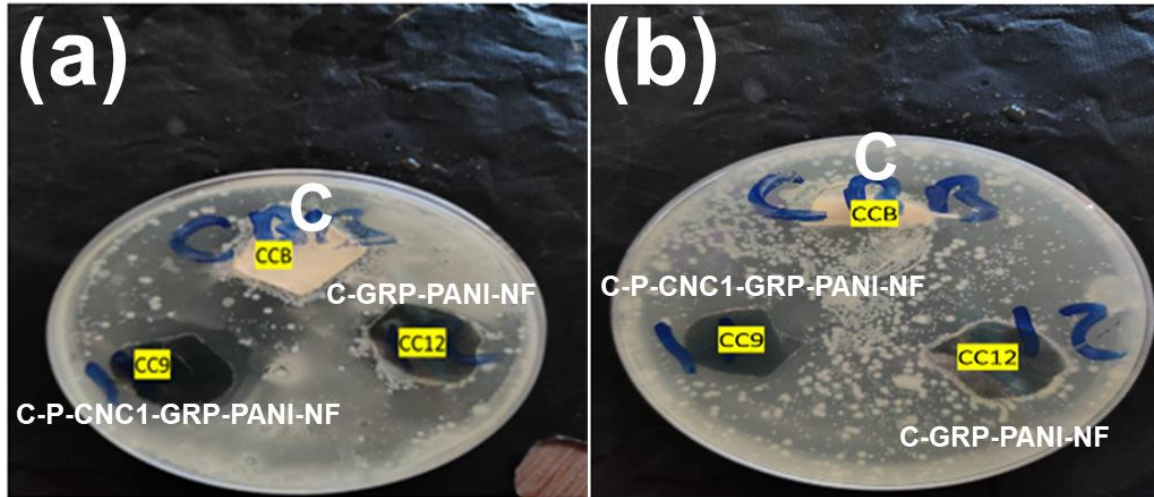


Fig. 5. Digital photos of clear inhibition zone of blank their different composites (a) *Staphylococci* bacteria and (b) *E-coli* bacteria

4. Conclusion

Multifunctional coating composites were prepared and applied on cotton fabrics. The smart coating was prepared from cellulose nanocrystals, conducting polymers and graphene, yielding uniform dispersion of coatings. The developed coated samples achieved a high rank in flame retardancy, with zero(mm/min) burning rate compared to 115 (mm/min) for the blank one. Treated fabric after the treatment process recorded significant improvement in antibacterial activity compared to the untreated sample recording a 35 mm clear inhibition zone compared to zero for blank. Additionally, the electrical conductivity was significantly enhanced to reach 12 K Ω .

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