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Fabrication of Graphene Coated Nonwoven Textile for Industrial Applications

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A cost effective electrically conductive textile for large scale applications will revolutionise many industries. Herein, we demonstrate a novel processing approach to produce conductive textiles for industrial applications. The conductive nonwoven textile was successfully fabricated using a simple dip coating method. The polyester nonwoven was coated with liquid crystallite graphene oxide with subsequent non-toxic chemical reduction. The process is readily scalable. The graphene coated fabric has been characterized by electron microscopy as well as electrical, mechanical, thermal and abrasion resistance measurements. It was found that the electrical surface resistivity of the prepared polyester-graphene composite fabric was $330 \Omega/\square$. The electrical surface resistivity was 3 and 150 times lower than that of polypyrrole coated woven polyester fabric and graphene coated nonwoven fabrics which were published previously. The hybrid polyester-graphene textile prepared here should find application as high-performance geotextiles or as heating elements.

Introduction

Electrically conductive textiles have been using in numerous applications including healthcare, military, and for wearable electronics. They have also been used as heating elements in industrial and residential heating components such as carpet, automobile seats and de-icing of aircraft drive ways [1]. Using electrically conductive nonwoven as a heating application will have benefits over metal heating elements as they are free of corrosion and low manufacturing costs are flexible, can be formed in to required shape and are light weight. In addition, conductive textiles can be used in industrial applications such as electrokinetic geosynthetics (EKG) which is a class of material used to replace conventional electrodes in leak detection systems. The most commonly used electrodes are metals such as iron, steel or copper [2-4]. EKGs may be a single composition material that are electrically conductive or composite materials in which at least one element is electrically conductive. EKGs based on polymeric materials have some advantages over metal electrodes including corrosion resistance, being able to form in to variety of shapes and low cost. A low cost electrically conductive nonwoven that can be produced on a large scale with easy installation will provide a more affordable mechanism over the above mentioned EKG materials.

Numerous studies have been reported for textiles modified with different types of conductive materials for smart textiles and heat generation applications. Most research has focused on either

coating or blending of polyester fibres or yarns with conductive materials [5-14]. The methods used to incorporate conductive materials with the fibres or yarns needs sophisticated processes and/or equipment which compromises large scale production. In contrast, process complications can be reduced considerably by incorporating conductive materials directly onto preformed textiles fabrics using polymerisation, deposition of metals, weaving or integration of metal fibres or wires [1, 13, 15-21].

The electrical conductors have been incorporated to textiles via printing or coating to get lightweight conducting materials. Printing or coating techniques can avoid high temperature gradients in heating elements as the temperature generated is distributed over the surface of the fibre composites [20, 22]. Further, the dip coating methods are attractive because of their similarity to dyeing processes already used in industry. Recently, the fabrication of a conductive polyester nonwoven was reported with a one-step dyeing like approach using an aqueous dispersion of reduced graphene oxide (rGO) using polyurethane as the adhesive layer to give a minimum surface resistance of $5 \times 10^4 \Omega/\square$ [23]. A similar approach is used in the current work.

The physical properties, commercial availability and easy processability are the major factors to be deliberated in large scale applications. Polyester is named to be one of the "greatest" man-made fibres and widely used in many hybrid composite applications due to its outstanding physical properties [24-26]. The mechanical properties, durability, abrasion resistance and resistance to weather are major characteristics of polyester fibres. Polyester fibres are readily converted to nonwoven textiles [27, 28]. This

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involves wet-laid [29] and melt-blown [30] web forming processes bonded with mechanical methods as needle punching, chemical or thermal bonding [31-34]. Nonwoven polyester textiles are strong, permeable, resistant to stretching heating, shrinking, abrasion, mildew and most chemicals, which enabled it to be used in applications such as filtering, geotextiles, thermal insulation, disposable hygiene products as surgical gowns, diapers and in many other areas [35-40]. Moreover, the thermal, mechanical and optical properties made graphene a choice of material to be used for wide variety of applications [41-46]. Graphene oxide (GO) produced by chemical method, can be manufactured in large scale [47-49]. The aim of this study is to investigate methods to generate high conductivity, abrasion resistant coatings on non-woven fabric using a dip coating method. Here in, we report a simple method of fabricating conductive nonwoven coating with liquid crystalline graphene oxide (LCGO) solution followed by chemical reduction for large scale industrial applications.

Materials and Methods

Preparation of LCGO

GO is prepared from the intercalated graphite flakes (3772, Asbury Graphite Mills USA). Typically the intercalated graphite flakes were first thermally expanded at 700 °C under the nitrogen atmosphere for less than ≤ 5 sec. The resultant expanded graphite (270-300 times volume expansion) was used as the precursor for GO synthesis. Typically, 10 g expanded graphite and 1 L of sulphuric acid were mixed and stirred in a flask using a mechanical stirrer and the temperature was reduced using an ice-salt bath. After 15 mins of stirring, 45 g of KMnO_4 was slowly added over a $\frac{1}{2}$ hr period. The slow stirring was continued for 3 hrs. The temperature was raised to 55 °C and maintained for 3 hrs. The reaction mixture became more viscous and the colour changed from green to grey. The contents were cooled with an ice bath for 30 minutes, and then 1 L Milli-Q water was added dropwise with continued ice-bath cooling. The addition of the initial 500 mL of Milli-Q water took approximately 90 minutes followed by the remained 500 mL which was added over 30 minutes. This procedure can be followed overnight depending on the requirement to keep the temperature low. The dropwise addition of 100 mL of 40% hydrogen peroxide (H_2O_2) solution changed the colour to bright yellow. The contents were further diluted by 10 L of Milli-Q water and allowed to settle overnight. The clear supernatant acid was decanted and 500 mL of diluted hydrogen chloride ($\sim 10\%$) was added to the reaction mixture for removal of manganese salts. The contents were stirred for 30 minutes and left to settle. The washing process was repeated for 2-3 times until the pH of the supernatant is ~ 2 . The contents were finally dialysed until the pH of washing water was ~ 3.4 . The GO as a viscous dispersion was obtained in 130-145% increase in weight based on graphite [47, 48, 50]. The two different concentration dispersions with LCGO content 0.25% wt and 0.6%wt were prepared.

Preparation of conductive nonwoven

The polyester nonwoven textile with liner density of 180 g/m^2 supplied by Bidim Nonwoven Geotextile-Australia, was cleaned in purified water via ultra-sonication. For the first coating cycle, the pre-treated nonwoven material was dipped in to the LCGO solution for 1-2 minutes and then removed. Then the LCGO dip coated textile was dried in the air at room temperature for 24 hrs. Subsequent coatings were applied using the same procedure up to 6th coating cycle. The figure 1 outlines the preparation method used. The amount of GO coated on to the nonwoven was controlled by changing the number of coating steps and by using different LCGO concentrations. Nonwoven with different number of coatings was prepared by dipping in to LCGO-0.25% wt and LCGO-0.6% wt.

Reduction of GO

The chemical reduction has been carried out to convert GO to reduced graphene oxide. The obtained LCGO coated nonwoven samples were chemically reduced after drying overnight by using 5 g of sodium dithionite and sodium bisulfite mixed in 4:1 ratio and dissolved in 900 ml of water. The reduction process was carried out at 95 °C for 1 hr. Next reduced graphene oxide coated nonwoven specimens (rGO) were washed in Milli-Q water to remove any remaining salts. The rGO samples were designated as per Table 1.

Table 1: Preparation of LCGO coated nonwoven samples

LCGO (%) in coating solution	Number of coating cycles	Designation of rGO samples
0.25	X=1,2,3,4,5,6	R' _X
0.6	X=1,2,3,4,5,6	R _X

Characterisation

X-ray Photoelectron Spectroscopy

The X-ray Photoelectron Spectroscopy (XPS) has been carried out to evaluate the graphene coating and chemical reduction. XPS is a spectroscopic surface sensitive technique that measures the composition of a material at the part per thousand ranges. The material is irradiated with an X-ray beam while simultaneously measuring the number of electrons that escape and their kinetic energy. XPS was conducted using a SPECS PHOIBOS 100 Analyser installed in a high-vacuum chamber with the base pressure below 10–8 mbar, X-ray excitation was provided by Al K α radiation with photon energy $h\nu = 1486.6 \text{ eV}$ at the high voltage of 12 kV and power of 120 W. The XPS binding energy spectra were recorded at the pass energy of 20 eV in the fixed analyser transmission mode. Analysis of the XPS data was carried out using the commercial CasaXPS2.3.15 software package. The LCGO coated nonwoven material after 3rd coating (GO₃) and rGO samples (R₁, R₂, R₃) were analysed for elemental compositions.

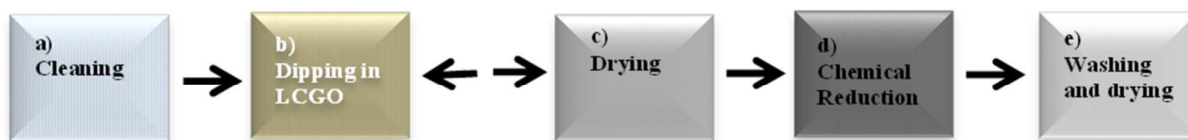


Figure 1: Representation of fabrication of rGO samples. a-cleaning in purified water, b-dipping the nonwoven in LCGO solution, c-drying in air, e-washing and drying to remove excess GO, d-chemical reduction. The number of coating cycles can be increased by repeating of processes “b” and “c”.

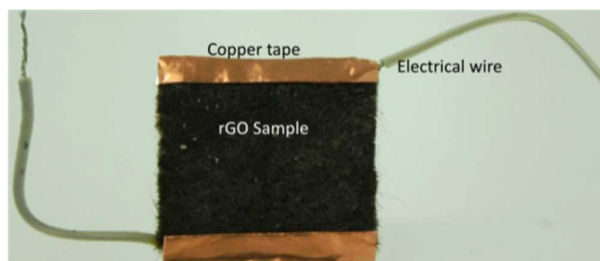


Figure 2: A photographic image of rGO sample connected with insulated electrical wires for characterisation of electrical properties during bending.

Electrical Properties

The effect of mechanical bending on the electrical resistance of rGO coated specimens was investigated with samples of 20 mm x 20 mm as shown in Figure 2. The electrical resistivity were characterised during and after bending. The deformation was carried out on a Shimadzu EZ mechanical tester with deformation rate of 20 mm/min and stroke of 9 mm for fifty bending and release cycles. The multimeter was connected to the specimens during the deformation test to continuously monitor the electrical resistance.

The surface resistivity of the samples was determined from.

$$\sigma_s = \frac{R \cdot W}{L}$$

where resistance R is measured using multimeter, σ_s is the surface resistivity, W is the width and L is the length.

Thermal Behaviour

The thermal characteristic of R₃ sample was evaluated by an infrared camera. The sample size was 200 mm x 200 mm and the sample was mounted to a frame with nonconductive strings. Then the two insulated wires were attached to R₃ using copper tapes, which were connected to a power source via alligator clips. The R₃ sample was heated by applying a voltage of 32.5 V (DC). The thermographic camera was used for infrared video capturing with the arrangement shown in Figure 3.

Morphological Observation

The surface morphology of rGO sample (R₃), LCGO coated nonwoven and raw nonwoven was observed by scanning electron microscope (SEM) of model of JEOL JSM -7500 FA. The surfaces of the samples were coated with platinum before analysis. In addition, the optical images of rGO samples with different number of coating cycles (1st, 2nd, 3rd and 6th coating) were captured by optical microscope (model LECIA M205 A) to observe the difference in GO deposition in nonwoven textile.

Abrasion Wear Resistance

The abrasion resistance was conducted on a 90 mm diameter circular sample of R₃. The sample was uniformly placed under one kilogram as an abrading load and the rotary stage containing the sample was rotated at 100 rpm for 1 hr. The sample was abraded against high-density polyethylene (HDPE) sheet supplied by bidim nonwoven geotextile-Australia. The resistance was measured after every 1 hr and the resistance change was calculated.

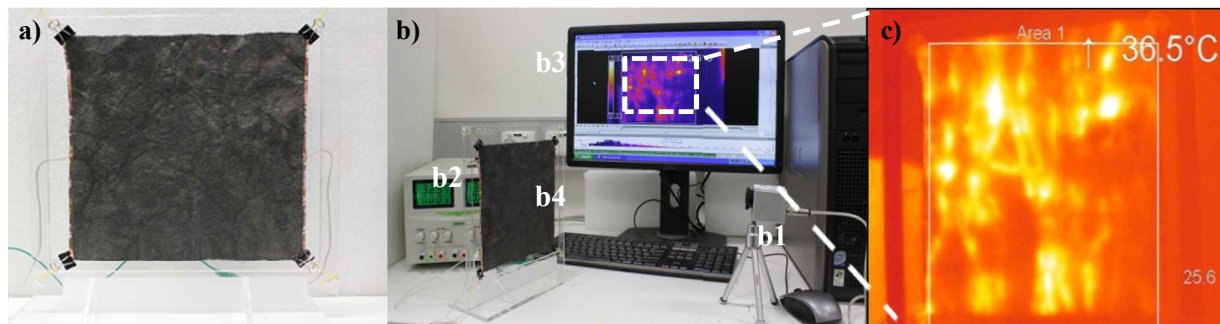


Figure 3: (a) As-Prepared graphene coated textile sample (R₃), (b) apparatus arrangement including b1-infrared camera, b2-power source, b3-screen and b4-rGO sample, (c) heating of rGO nonwoven (R₃) prepared using 3 coating cycles of 0.6wt% of LCGO.

Results and Discussion

Graphene Oxide Coating

The percentage weight increment of the samples is illustrated in Figure 4 for successive coating cycles. As can be seen in the graph the coating mass increases approximately linearly with the number of coating cycles. The amount of GO adsorbed onto the surface of the fabric increased when a more concentrated LCGO solution was used. The GO adsorption to the fibre surface is influenced by the concentration of LCGO, fibre surface tension and the surface area of the fibres. The chemical interaction between the nonwoven and GO is a result of van der Waals forces, hydrogen bonding between the atoms of the ester groups of polyester fibres and the anionic functionalities of LCGO [51].

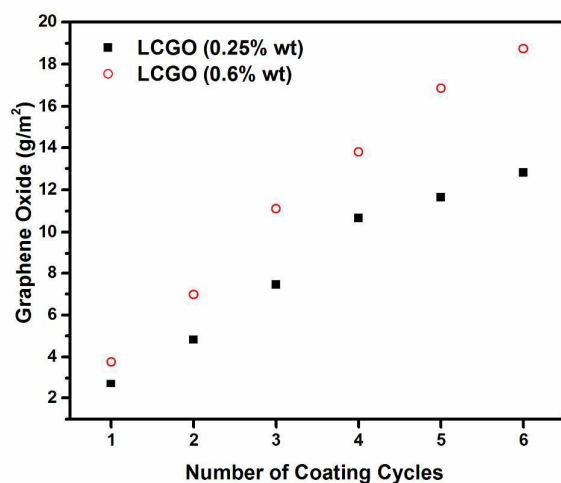


Figure 4. GO deposition on to nonwoven as a function of coating cycle for two different coating solutions having the indicated concentration of LCGO.

XPS

The elemental analysis was carried out for nonwoven samples to investigate the nature of the coating and chemical reduction processes. The chemical process was used to reduce the oxygen containing groups, in order to convert GO into rGO. The rGO is a chemically improved version of graphene with a conjugated structure that modify the thermal and electrical properties. As can be seen in Table 2, the percentage of carbon increases with the number of coating cycles. This further confirms the GO mass increment with successive coating cycles. In addition, the percentage of oxygen decreased approximately by 17% for the highest conductive sample (R₃) which was obtained after the chemical reduction of GO₃, due to the removal of oxygen containing groups.

Morphological Observations

The surface morphology of as-received nonwoven fabric, GO coated and rGO samples are shown in Figure 5 (a-c), respectively. As can be seen in Figure 5(a), fibres are randomly distributed within the

nonwoven material. The absorbed LCGO is encapsulated in inter fibre porous areas, around the individual fibres as presented in Figure 5 (b) and (c). Polyester fibres are inherently hydrophobic, due to the lack of polarity groups. The hydrophobic property of the polyester fibre counterattacks the entry of water molecules into the interior of fibres. Therefore, it can be assumed that the amount of LCGO absorbed into the fibre interior is minimal. As can be clearly seen in Figure 5(d), GO is deposited in-between interstices of fibres. The Figure 5(e) is presenting the rGO layers in the fibre composite. The dimensions of the interstices, the number of interstices per unit mass of the nonwoven, regional variations in fibre packing density will influence the GO absorbed to the nonwoven composite. The low magnification photographic view of as-prepared conducting nonwoven clearly shows the uniformly coated fabric structure (Figure 5(f)).

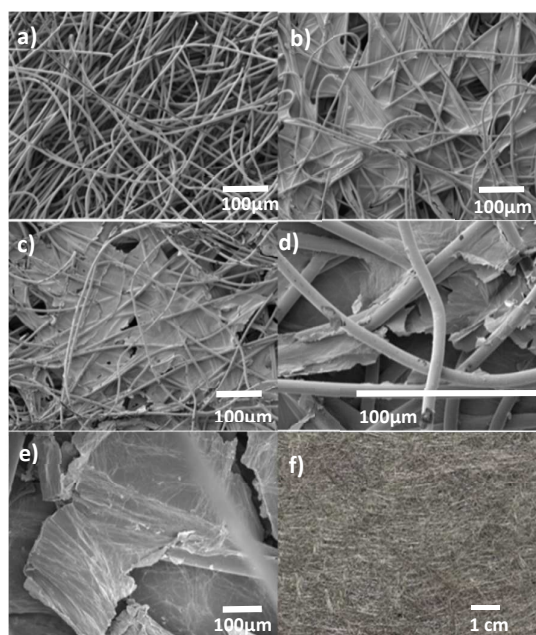


Figure 5: SEM images of (a) raw nonwoven (b) LCGO coated nonwoven (c) rGO sample (d) rGO in to interstices of fibres (e) rGO layers (f) photograph of as-prepared conducting graphene nonwoven.

Table 2: XPS data

Sample	Mass of elements (%)	
	Carbon(C)	Oxygen(O)
1st coating reduced (R1)	77.22	22.78
2nd coating reduced (R2)	81.75	18.75
3rd coating reduced (R3)	83.56	16.44
3rd Coating non reduced (GO ₃)	66.87	33.13

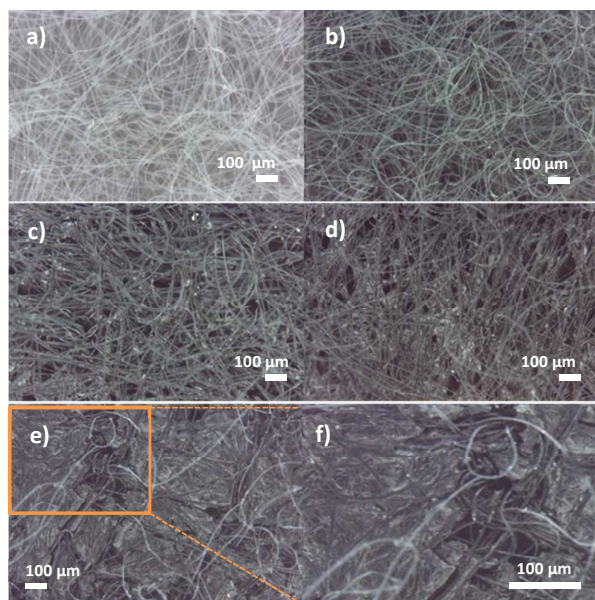


Figure 6: Optical microscope images of as prepared samples; (a) raw nonwoven, rGO sample after (b) 1st, (c) 2nd, (d) 3rd e) 6th coating cycles and f) higher magnification.

The optical microscope images of raw nonwoven and rGO samples after 1st, 2nd, 3rd and 6th coating cycles are presented in figure 6 (a-f). As can be seen in optical images after the 3rd coating cycle most of the interstices in nonwoven are covered by the GO. Therefore, it can be mentioned that more GO layers are deposited in surface of textile forming thicker GO layers after 3rd coating cycle which has less interaction with fibres in nonwoven as seen in figure 6(e).

Electrical Properties

The surface resistivities of the samples were calculated to analyse the relationship of the electrical resistivity to the number of coating cycles. The surface resistivity of rGO samples and the percentage weight of GO added to the nonwoven are presented in Figure 7 as a function of coating cycles. It was found that the lowest surface resistivity which is 330 Ω/\square was reported after the 3rd coating cycle in LCGO 0.6% wt. The rGO categories exhibited the enhanced electrical conductivity values with the increment of GO add-on up to the 3rd coating cycle and from the 4th coating cycle samples exhibited a nonlinear behaviour in surface resistance with increasing amount of GO added to the fabric. The possible major factor of high resistivity after the first coating cycle even with the higher concentration of LCGO is the disconnections within the fibre matrix which reduced the conductive pathways. The increasing number of dip coating cycles after the 3rd coating and subsequent reduction under the identical conditions were not evolving any further improvement in surface resistance. As a result of more GO deposition on the surface of the nonwoven fabric caused less interaction of LCGO with polyester fibres. During the reduction

process the less interacted and unstable rGO layers to delaminate from the samples so that small amount of reduced GO remained in the nonwoven textile. Further, the thick rGO layers with less bonding to the fibres have a high chance of separating from the sample. Therefore, more GO add-on to the nonwoven textile has not contributed to a decrease in the electrical resistivity due to the instability of the rGO layers within the fibre matrix. The value of maximum conductivity reported in this study is 150 fold enhance the surface conductivity of the rGO deposited polyester nonwoven reported in the literature[23].

The optimum condition for as prepared nonwoven is with 6% and 4% GO addition to the textile with LCGO concentrations of 0.6% wt and 0.25% wt respectively. To evaluate durability of as-prepared samples, the bending-electrical resistance performance of the rGO nonwoven textiles was measured. The resistance change during fifty bending cycles was monitored for groups of samples in order to understand the material behaviour during and after bending. The change in the electrical resistance as a fraction of the initial resistance ($\Delta R/R_0$) for rGO samples are shown in Figure 8. The conducting nonwoven samples were subjected to repeated bending-relaxation to 90° up to 50 cycles. These samples suffered 1.14% maximum change in the average $\Delta R/R_0$ when bent to 90°. In addition, all the samples exhibited a stable resistance change during bending for 50 consecutive cycles.

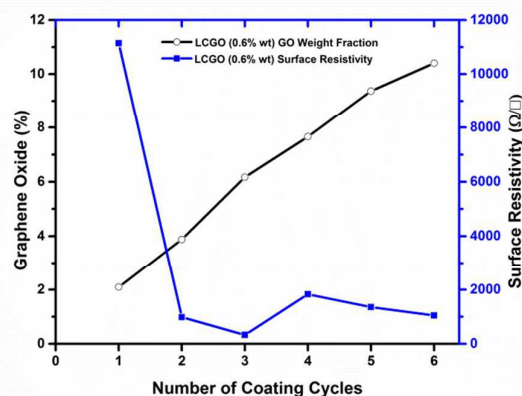


Figure 7: Surface resistivity and % add-on of GO as a function of coating Cycles of LCGO (0.6% wt)

Thermal Behaviour

The thermal behaviour of the fabric was analysed in order to confirm the capability of the material in heating applications. The fabric measures 200 mm \times 200 mm with 6 wt % of GO was heated up to average temperature of 36°C with 32.5 V and 0.05 A within 10 min. The thermal behaviour of the sample exhibited an considerable heating capacity as the textile heating element published in the literature with 44.3% wt add-on of polyethylenedioxythiophene/p-toluene sulfonic acid [1].

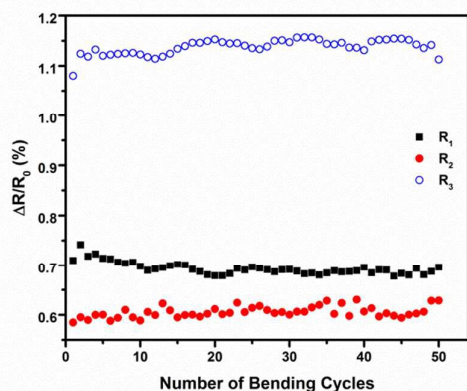


Figure 8. The average normalised resistance change of rGO-0.6wt% specimens during fifty bending cycles. Resistivity values R_1 , R_2 , and R_3 are for rGO nonwovens with 1st, 2nd and 3rd coating respectively.

Abrasion Resistance

Abrasion resistance of the textile materials is very complex phenomenon and affected by many factors, mainly classified as fibre, yarn, fabric properties and finishing processes. Some of these parameters affect the fabric surface whereas some of them have an influence on internal structure of the fabrics. For example fibre characteristics like composite ratio and fineness play a significant role in surface abrasion, while yarn and fabric characteristics like yarn linear density and interlacing coefficient are significantly related with structural abrasion. The mechanical properties and dimensions of the fibres are important for abrasion. Fibre type, fibre fineness and fibre length are the main parameters that affect abrasion. The abrasion resistance of the nonwoven sample was evaluated in multi directions by rotating the nonwoven sample against a fix load abrading with a geo-membrane. The electrical resistance was increased by 20 % after 6000 abrading cycles. The percentage increment of the electrical resistance is well below the 200% percentage change in the electrical resistance mentioned in literature where the resistance change from $10 \Omega/\square$ to $30 \Omega/\square$ after 10000 cycles[1].

Conclusion

The above analysis justifies the successful combination of polyester nonwoven with graphene oxide from a simple dip coating technique. The surface electrical resistivity of $330 \Omega/\square$ and self-heating up to 36°C within 10 minutes can be considered as well suited to many industrial applications. The average resistance changed minimally during bending. The abrasion resistance test with 20% electrical resistance change after 6000 abrading cycles further confirms the usability of the conductive nonwoven in industrial applications. Furthermore, by considering the morphological observations it can be mentioned that nonwoven is the most appropriate material to be used in conductive coating

process, especially when dealing with hydrophobic fibres as majority of GO layers are encapsulated between interstitial regions of textile. Polyester fibres are inherently resistant to climatic changes and polyester nonwoven has already been using in geo-textile applications. The prepared textile can be used as EKG in leakage detection surveys, due to the electrical conductive properties in comparison with EKG materials discussed in the introduction. In addition, due to the heating property this can be used as a heating component for both industrial and residential applications such as floor carpets, cars seats and can be further improved for de-icing of aircraft driveways. The LCGO coated nonwoven material can be proposed as a manufacture feasible, flexible, free of corrosion and light weight superlative material which will open up new avenues in conductive textiles.

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A hybrid electrically conductive polyester-graphene textile was fabricated as high-performance smart textile for geotextiles and/or heating elements applications.

