



## Fabricating electroconductive cotton textiles using graphene



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### ABSTRACT

In this work, design parameters were investigated for enhancing the conductivity of graphene-coated cotton textiles. Graphene oxide (GO) was immobilized on cotton fabric through a conventional “dip and dry” method. The GO-coated fabrics were then immersed in an aqueous solution of reducing agent, which converted the GO into graphene. The effect of various parameters such as type of reducing agent (NaBH<sub>4</sub>, N<sub>2</sub>H<sub>4</sub>, C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> and NaOH) and its concentration, reduction time and number of coating process on conductivity of the fabrics was studied. The mechanical performance of the fabrics was also investigated. The results showed that the best conductivity and mechanical performance were obtained using Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> as a reducing agent. The reaction time of 30 min at 95 °C was enough for complete reduction of the GO. Electrical conductivity increased by approximately three orders of magnitude with the increase in the number of coating process from 1 to 20 cycles.

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### 1. Introduction

Flexible fibrous textile materials containing conductive fillers have recently attracted much attention for their use in a wide range of applications, especially in advanced composites and E-textiles (Cakir, 2011; Cherenack, Zysset, Kinkeldei, Münzenrieder, & Tröster, 2010; Mattila, 2006; Robert, Feller, & Castro, 2012; Shim, Chen, Doty, Xu, & Kotov, 2008; Tao, 2001; Yang, Lightner, & Dong, 2011). E-textiles are fabrics that enable computing, digital components and electronics to be embedded in them. Similar to classical electronics, construction of electronic capabilities on textile fibers requires use of conducting and semi-conducting materials. Electroconductive textiles can be made using metal strands or metallic fibers mixed with textile fibers woven into the construction of the textile. However, because both metals and classical semiconductors are stiff material, they are not very suitable for textile fiber applications since fibers are subjected to a high degree of stretch and bending during their application. There is also an interest in semi-conducting textiles, made by impregnating normal textiles with intrinsically conducting polymers, carbon black, carbon nanotubes or metal-based powders (Babu, Dhandapani, Maruthamuthu, & Kulandainathan, 2012; Egami et al., 2011; Havel, Behler, Korneva, & Gogotsi, 2008; Little, Li, Cammarata, Broughton, & Mills, 2011; Negru, Buda, & Avram, 2012; Zhao, Cai, Zhou, & Fu, 2011). But, the most widely used methods require complex processes, expensive

materials and pre-functionalization and have some disadvantages such as lacking uniform coating, flexibility and durable wear resistance, which increase cost of production.

Graphene is a flat monolayer of carbon atoms which is tightly packed into a two-dimensional honeycomb lattice and is a basic building block for graphitic materials of all other dimensionalities (Geim & Novoselov, 2007). Due to its outstanding electronic, optical, excitonic, thermal and mechanical properties, graphene differs from most conventional three-dimensional materials and has attracted great interest as an outstanding candidate for the production of advanced materials with many potentials in various applications (Allen, Tung, & Kaner, 2010; Bai & Shen, 2011; Huang et al., 2011; Rao, Sood, Subrahmanyam, & Govindaraj, 2009; Zhu et al., 2010). Graphene is more suitable for E-textiles because it can be conducting and semiconducting and applied as a dye to make electrically conducting textiles (Dong et al., 2012; Fugetsu, Sano, Yu, Mori, & Tanaka, 2010; Khan, Young, O'Neill, & Coleman, 2012; Shin et al., 2012; Yu et al., 2011). It has been demonstrated that aqueous dispersions of graphene can be readily produced without the need for polymeric or surfactant stabilizers due to the presence of carboxylic and hydroxyl groups. These advantages lead to the possible direct application of graphene in preparing electroconductive textiles using the same strategy that has been used to make textile finishing through simple dip-pad or dyeing assembly.

Graphene can be prepared in large quantities through chemical conversion from graphite, which has facilitated fabrication of graphene-based materials. Graphene oxide (GO) has been prepared by harsh oxidation using Hummer's method (Hummers & Offeman, 1958). The as-made GO obtained in this way is electrically insulating due to the attached oxygen functional groups. Consequently,

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various reduction methods such as thermal or chemical reduction have been developed to restore its electrical conductivity. Thermal reduction has been highly effective in producing graphene-like films. However, this approach is limited by the choice of substrate; i.e., typical textile substrates cannot be used due to their low degradation temperature. On the other hand, chemical reduction can be realized at the temperature lower than 100 °C, which is extremely important for practical applications. Many reducing agents with generally low cost have been developed by a simple method to reduce GO; but, the reduction mechanism by chemical reducing agents has been unclear and the electrical conductivity of the graphene film is very different. Therefore, it is necessary to develop a more effective chemical reduction method to produce graphene-based materials with high electrical conductivity.

In this paper, the fabrication of electroconductive cotton textiles was explored using graphene. GO was deposited on the surface of fabric through a conventional “dip and dry” approach. The GO loaded fabrics were then chemically reduced for conversion into the electroconductive graphene. The chemical reduction of GO with several reducing agents was examined to investigate their effect on conductivity of the fabric. Effect of reductant concentration, reduction time and the number of coating cycles were also investigated. Although numerous studies have investigated effect of reducing agent on electrical properties of graphene materials, this is the first report on the effect of reducing agent on electrical properties of graphene-coated textiles. The results are important for understanding and controlling electrical properties of graphene-coated textiles and their possible applications.

## 2. Experimental

### 2.1. Materials

Graphite purum powder (particle size < 100 μm) was purchased from Fluka. All other materials including hydrazine (N<sub>2</sub>H<sub>4</sub>), sodium borohydride (NaBH<sub>4</sub>), sodium hydrosulfite (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>), sodium hydroxide (NaOH), ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>), potassium permanganate (KMnO<sub>4</sub>), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 35%), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 98%) and hydrochloric acid (HCl, 37%) were supplied by Merck. Desized, scoured and bleached 100% cotton fabric was used as the substrate. The water used in all the experiments was purified using a Milli-Q system with the resistivity of higher than 18.2 MΩ cm<sup>-1</sup>.

### 2.2. Synthesis of GO

GO was synthesized from graphite powder by the modified Hummer's method (Hummer & Offeman, 1958). One gram of as-purchased graphite powder was added to 25 ml of H<sub>2</sub>SO<sub>4</sub> (98%) and was left while stirring for 12 h. Afterwards, while keeping the temperature at less than 10 °C, 3.5 g of KMnO<sub>4</sub> was added. This was stirred at 50 °C for 2 h. Next, 70 ml of water and 5 ml of H<sub>2</sub>O<sub>2</sub> were added and then the mixture was stirred for 30 min. For purification, the resulting mixture was centrifuged, rinsed first with 5% HCl and then three times with water. 200 ml of water was added to the resulting product to make 0.5% (w/w) dispersion. This 0.5% aqueous dispersion was used to prepare all of the subsequent dispersions for deposition.

### 2.3. Preparing GO-coated cotton fabric

Aqueous dispersion of GO with the concentration of 0.05% was prepared and bath-sonicated for 60 min. The cotton fabric was dipped into the prepared dispersion, soaked for 30 min at room temperature and then dried at 90 °C for 30 min. Because of the strong adsorption, the fabric was quickly coated by the GO. The

coating process was repeated three times in order to increase GO adsorption.

### 2.4. Preparing graphene-coated cotton fabric

The GO-coated samples (1 g of each) were immersed in 100 ml aqueous solution of 25 mM reducing agents of NaBH<sub>4</sub>, N<sub>2</sub>H<sub>4</sub>, C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> and NaOH. The mixture was kept at 95 °C for 60 min under constant stirring. The resulting fabric was washed with a large amount of water several times to remove the excessive reducing agents. At the end, the samples were dried at 90 °C for 30 min. The resulting graphene-coated fabrics were referred to as NaBH<sub>4</sub>-GO-cotton, N<sub>2</sub>H<sub>4</sub>-GO-cotton, C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>-GO-cotton, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>-GO-cotton and NaOH-GO-cotton based on the used reducing agents.

### 2.5. Characterization

Transmission electron microscopy characterization of the GO nanosheets was performed using a microscope LEO 912AB. A droplet of GO dispersion was cast onto a TEM copper grid and the solvent was evaporated overnight at room temperature. Surface morphology of the fabrics was examined by a KYKY-EM3200 scanning electron microscope after sputter coating with a very thin layer of Au. The ultraviolet-visible (UV-vis) reflectance spectra of the fabrics were recorded on a Lambda 35 UV-vis spectrometer (Perkin Elmer Instruments Co. Ltd., USA). Color coordinates were determined by color measurement software using the CIE L\* a\* b\* color space at D65/10°. Electrical surface resistivity of the fabrics was measured using a standard two-probe method (Petersen, Helmer, Pate, & Eichhoff, 2011) by means of Sa-Iran digital multimeter model 8515. Tensile strength and elongation of the samples were evaluated using a MESDAN LAB instrument according to the standard test method of ISO 5081 (strip method). The constant cross-speed of approximately 50 mm min<sup>-1</sup> was used throughout the experiments. The measurements were performed in the warp direction of fabrics and average values were obtained from measurements of four samples. The amount of GO deposited on fabric was determined by reweighing fabrics after drying for 1.5 h in an oven at 80 °C. An analytical balance (Scatec SBA32) with 10<sup>-4</sup> precision was used to measure the samples' weights. Abrasion resistance was measured according to ASTM D4966-98. The end point was reached when two yarns were broken.

## 3. Results and discussion

Since cellulosic substrates have low stability at high temperatures and degrade quickly, using thermal methods, which produce graphene with high electrical conductivity, is not possible for the reduction of GO-coated cotton textiles. On the other hand, despite the possibility of graphene preparation via thermal reduction methods, this method cannot be used not only because of the limitations such as high price and nanosheets aggregation but also for low adsorption of graphene to cellulosic fabrics (lack of surface functionality and hydrophobicity nature of graphene). Therefore, it seems that covering the fabrics with GO and subsequently reduction treatment through low-temperature chemical methods can be a highly effective method.

GO was synthesized from natural graphite using Hummer's method that derivatize graphene sheets with carboxyl, carbonyl, hydroxyl and epoxide groups, thereby, breaking the p-conjugation in the two-dimensional carbon networks (Li et al., 2011; Park et al., 2008). The resulting product of GO powder was water dispersible, insulating and light brown in color. Fig. 1 shows TEM micrograph of a typical GO nanosheet deposited on a standard TEM grid. The sheet was several micrometers in dimension with the wrinkled (rough)

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