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Optimized preparation of electrically conductive cotton fabric by an industrialized exhaustion dyeing with reduced graphene oxide

Weifeng Shen · Yongchun Dong · Guixin Cui · Bing Li

Abstract Cotton woven fabrics were first dyed with a graphene oxide (GO) aqueous dispersion using a simple industrialized exhaustion dyeing process. The resulting fabrics were then chemically reduced to prepare the electrically conductive cotton fabrics. They were characterized by the FE-SEM, XPS and Raman spectrum and evaluated with respect to whiteness, electrical surface resistance and electrochemical impedance spectroscopy. Some factors affecting the process such as the GO concentration, dyeing and reducing conditions, and nature and concentration of the reducing agent were also investigated. The results indicated that GO could be fixed on cotton fabric by a simple exhaustion dyeing process and converted into the reduced GO (RGO) with a reducing agent. Increasing the GO concentration or dyeing temperature and time decreased the whiteness and electrical surface resistance of the RGO-dyed fabric. However, higher pH showed a reverse effect. $Na_2S_2O_4$ was found to be a stronger reducing agent for the conversation of GO into RGO on cotton fabric than ascorbic acid and thiourea. The reduction of GO-dyed fabric was easily completed by an increasing $Na_2S_2O_4$ concentration at higher temperature. Moreover, an increasing number of dyeing cycles decreased the surface resistance and impedance modulus and increased the abrasion resistance of the RGO-dyed cotton fabric.

Keywords Graphene oxide · Cotton fabric · Exhaustion dyeing · Reduction · Electrically conductive

W. Shen · Y. Dong (✉) · G. Cui · B. Li
Division of Textile Chemistry and Ecology, School of Textiles, Tianjin Polytechnic University, 399 Bingshui West Road, Xiqing District, Tianjin 300387, People's Republic of China
e-mail: teamdong@sina.cn;
dye@tjpu.edu.cn

Y. Dong
Key Laboratory of Advanced Textile Composites, Ministry of Education, Tianjin Polytechnic University, Tianjin 300387, People's Republic of China

G. Cui
Jiangnan Branch, China Textile Academy, Shaoxing 312071, People's Republic of China

Introduction

Electronic textiles and wearable electronics are innovative textile materials, which generally include high-performance sportswear, wearable displays, portable power and embedded health monitoring devices, etc. They incorporate new classes of electrically conductive fibers with an array of novel functionalities, such as flexibility, stretchability and being lightweight, directly into the textile itself (Liu et al. 2012). It is well known that electrically conductive textiles can be prepared using metallic fibers mixed with textile fibers woven into the construction of

the textile. Conductivity of fabrics has been also obtained by coating with the conjugated polymers, carbon black, carbon nanotubes, metal-based powders or indium tin oxide (Shaterikhalilabad and Yazdanshenas. 2013b). However, these technologies are not cost-effective for commercial production because they often contain expensive materials and complicated processes. On the other hand, a few attempts (Kaempgen et al. 2009; Pushparaj et al. 2007; Hu et al. 2011; Jost et al. 2011; Liu et al. 2012; Yu et al. 2011) have been made to integrate nanoscale materials into textiles to prepare flexible, thin and lightweight power conversion and storage devices.

Graphene as a two-dimensional, single-layer sheet of sp2 hybridized carbon atoms has attracted a great deal of attention because of its high electronic conductivity, good thermal stability, and excellent mechanical strength (Geim and Novoselov 2007; Dong et al. 2012). Furthermore, it has been used on cotton (Liu et al. 2012; Shaterikhalilabad and Yazdanshenas 2013a, b), polyester (Molina et al. 2013a) and acrylic (Fugetsu et al. 2010) textiles as an electronic conductive coating. Graphene is often generated using several methods such as chemical vapor deposition, micromechanical exfoliation of graphite, epitaxial growth on an electrically insulating surface and creation of colloidal suspensions by chemical reduction (Zhou et al. 2011; Molina et al. 2013a). The chemical reduction method is widely used owing to its simple procedure and low cost for the possible route for industrial production. More importantly, graphene converted with graphene oxide (GO) using a reducing agent is very suitable for textiles with low degradation temperature because chemical reduction can be realized at temperatures lower than 100°C (Shaterikhalilabad and Yazdanshenas 2013a). GO is heavily oxygenated graphene bearing hydroxyl and carboxyl groups and is highly dispersible in both aqueous and polar organic solvents (Fugetsu et al. 2010). It should be noted that GO has a similar structure to the vat dye used in the textile industry (Zhou et al. 2011). Therefore, GO sheets are fixed on the surface of different fibers using the conventional dyeing methods because of the attraction forces existing between them; subsequently, it is converted to graphene by the reducing agents used in the textile industry (Fugetsu et al. 2010; Zhou et al. 2011; Shaterikhalilabad and Yazdanshenas 2013a). Although the coating of graphene on cotton fabric for electronic conductivity has been reported in previous studies (Krishnamoorthy

et al. 2012; Liu et al. 2012; Shaterikhalilabad and Yazdanshenas 2013a; Samad 2014), the coating methods used were found to be hand operation or crude processes such as soaking (Shaterikhalilabad and Yazdanshenas 2013b; Sahito et al. 2015), brushing (Liu et al. 2012; Javed et al. 2014) or dipping (Krishnamoorthy et al. 2012; Samad 2014) without modern industrialized equipment, which may lead to an uneven and unfixed coating of RGO sheets on the cotton fiber surface, thus limiting their future commercial application. Additionally, some factors affecting RGO coating processes were investigated in the previous studies. However, the coating process parameters were not systematically optimized toward better electrical conductivity in the case of using industrialized equipment, which may reduce their practical importance. Thus, it is expected that production of large-scale RGO-coated cotton fabrics can be started by using industrialized processes and equipment for their increasing applications. In this present work, cotton woven fabric was selected to be dyed with GO aqueous dispersion using a simple industrialized exhaustion dyeing process because cotton fiber is a commonly used cellulose fiber in modern textile industries worldwide. It has a hierarchical network structure with a complicated surface morphology, hydrophilic groups and high porosity (Hu et al. 2010; Pasta et al. 2010; Li et al. 2015), which favor better adsorption of GO into cotton fiber from water than synthetic fibers. On the other hand, it is well known that the exhaust process is the most common and low cost method for dyeing textile materials. Moreover, textile substrates can be dyed in exhaust processes in almost any stage of their assembly into a textile product including fiber, yarn, fabric or garments (Perkins 2004). The GO-dyed cotton fabrics were then chemically reduced to prepare the electrically conductive fabrics. They were characterized by the FE-SEM, XPS and Raman spectrum and evaluated with respect to whiteness, electrical surface resistance and electrochemical impedance spectroscopy (EIS). Some factors affecting the process such as the GO concentration, dyeing and reducing conditions, and nature and concentration of reducing agent were also discussed. Consequently, the best dyeing and reducing conditions are demonstrated to optimize the electrical conductivity of the resulting cotton fabrics for the future industrialized applications.

Experimental section

Reagents and materials

Graphene oxide (GO) was purchased from The Sixth Element (Changzhou) Materials, Inc., China. Sodium dithionite ($Na_2S_2O_4$), thiourea (CH_4N_2S) and ascorbic acid ($C_6H_8O_6$) were of analytical grade and used as received. Commercially scoured and bleached cotton woven fabric (118.2 gm^{-2}) was used in this study. This fabric was further treated with a solution containing 1.0 g l^{-1} Na_2CO_3 and 2.0 g l^{-1} soap at a boil for 30 min, then thoroughly washed with cold water and dried at ambient temperature before use. Deionized water was employed throughout the study.

Dyeing of cotton fabric with reduced GO (RGO)

The GO aqueous dispersion was first prepared by mixing GO powders with the deionized water in an ultrasound bath for 30 min. A laboratory dyeing machine with 12 stainless steel dyeing tubes of 500 ml capacity (Datacolor International Applied Color System, Inc., USA) was used for dyeing. The known weight of the pretreated cotton fabric was dyed with different concentrations of GO aqueous dispersion at the appropriate temperature using a fabric/liquor ratio of 1:50 for a given time, and then the impregnated fabric was padded on a laboratory mangle (Mathis AG, Switzerland) with wet pick-up of 75–80 %. This procedure was repeated several times to increase the GO loading on cotton fabric. Afterward, 2.0 g of GO-dyed fabric was immersed in 100 ml aqueous solution containing varied concentrations of reducing agent at the design temperature for a certain time. Finally, the RGO-dyed fabric was rinsed thoroughly with warm deionized water and dried at 60 °C in an oven.

Characterization of RGO-dyed cotton fabric

Field-emission scanning electron microscopy (Hitachi S-4800, Japan) operating at 15 kV was used to observe the surface morphology of the RGO-dyed fabrics. Their binding energy analyses were performed on a PHI 5600 X-ray photoelectron spectrometer (Pekin-Elmer Inc., USA), and the binding energy of C1s was shifted to 284.8 eV as the reference. Raman spectra were recorded from 100 to 2700 cm; the fabric was further treated with a solution containing 1.0 g l^{-1} Na_2CO_3 and 2.0 g l^{-11} on a Renishaw 2000 Confocal Raman Microscope (Rhenishaw Instruments, England). Electrical surface resistance of the fabrics was measured through a standard two-probe method using a digital dual-display multimeter (Agilent U3402, USA). The measured distance of the fabric was 1.0 cm. An Alpha-A high-resolution dielectric, conductivity, impedance and gain phase modular measurement system with a ZGS active sample cell (Novocontrol BDS-50, Germany) was used to perform electrochemical impedance spectroscopy (EIS) analyses in the range of $10–10^7$ Hz for the measurement of the electrical resistance. Whiteness of the dyed fabrics was tested using a WSB-2 fluorescent whiteness meter (Beijing Kangguang Optical Instrument Co., China) since the color can be employed as a tool for easy detection and simple evaluation of the amount or thicknesses of graphene and GO multilayer systems (Inhwa et al. 2011). The dry and wet rubbing fastness of the RGO-dyed fabric was examined by a rubbing fastness tester according to the ISO international standard (ISO105X12/D02).

Results and discussion

Dyeing of cotton fabric with GO

In order to meet the demands for rapid and effective GO dyeing of cotton fabric in future commercial applications, the pretreated cotton fabric was dyed at different conditions using an exhaustion process, and then the dyed cotton fabrics were reduced with $Na_2S_2O_4$ for conversion into the graphitic-colored and electrically conductive fabrics. Their electrical surface resistance and whiteness were measured and shown in Fig. 1.

Figure 1a–c shows that the surface resistance and whiteness of the dyed cotton fabric gradually decreased with increasing GO concentration or dyeing temperature and time. However, an insignificant decrease in surface resistance was found when the GO concentration was over 0.50 %. This result suggested that cotton fabric was covered with GO sheets, thus leading to a high electrical conductivity. This is because GO sheets have various functional groups such as carboxyl, carbonyl and hydroxyl; epoxyl groups give them a hydrophilic nature (Si and
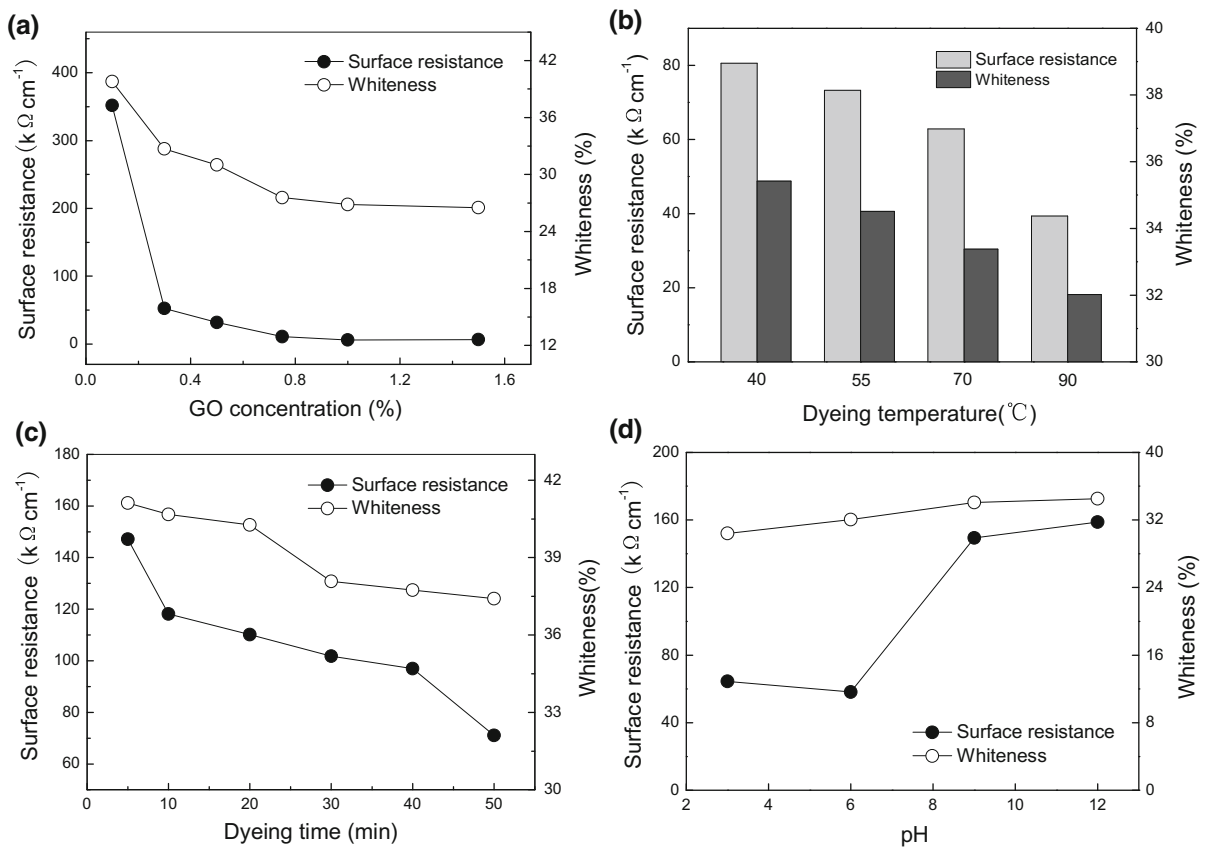
Fig. 1 Effect of the GO dyeing process on the surface resistance and whiteness of dyed cotton fabric

Samulski 2008; Krishnamoorthy et al. 2012). This allows GO sheets to be readily soluble in water at molecular levels with high surface affinity for cellulose fibers owing to the attraction forces such as van der Waals forces and hydrogen bonds between them (Shaterikhalilabad and Yazdanshenas 2013b; Molina et al. 2013b). Consequently, when cotton fabric was immersed in the GO aqueous dispersion, GO sheets were strongly adsorbed on the fiber surface to form the thinnest layer possible, with a thickness equal to a single GO (Li et al. 2008; Cote et al. 2009; Fugetsu et al. 2010). Moreover, increasing the GO concentration could enhance the contact of GO molecules with cellulose fibers in the solution, thus resulting in a significant number of GO molecules adhered to the cellulose fibers. GO sheets have a greater tendency to disaggregate at high temperature or long dyeing time. Besides, temperature elevation is also favorable to the degree of swelling of cotton fiber in the aqueous medium because of its low crystallinity and the

orientation of the molecular chains (Yi et al. 2012). On the other hand, Fig. 1d shows that higher pH increased the surface resistance and whiteness of the dyed cotton fabric. A reason for this is that cotton cellulose fiber usually exhibited a negative nature in the aqueous solution (Lim and Hudson 2004). GO is a heavily oxygenated graphene bearing hydroxyl and carboxyl groups (Fugetsu et al. 2010). Therefore, the GO molecule has low affinity for the cellulose fiber because of the enhanced charge repulsion between its ionized carboxyl groups and cellulose fiber at higher pH, especially in the alkaline pH range. On the contrary, weakening of the electrostatic interaction promoted GO molecules on the surface of the cellulose fiber at the lower pH level.
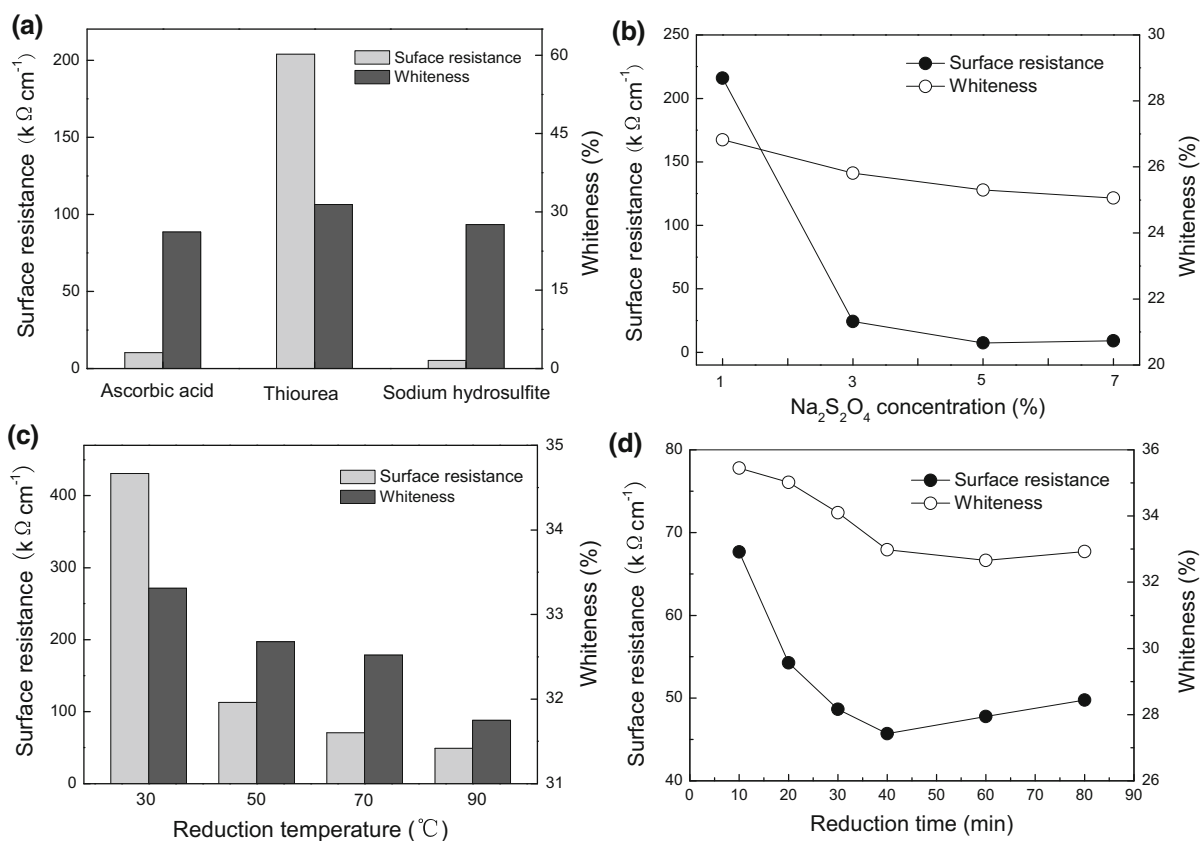
Reduction of GO-dyed cotton fabric

To select the most effective reducing agent and optimize the process variables for reduction of the

GO-dyed cotton fabric, three reducing agents including ascorbic acid, thiourea and $Na_2S_2O_4$ were used at different conditions to examine their influence on the surface resistance and whiteness of the RGO-dyed cotton fabric, and the results are presented in Fig. 2.

Figure 2a clearly shows that the GO-dyed cotton fabric reduced with $Na_2S_2O_4$ had a much higher electrical surface resistance than those with the other two reducing agents under the same conditions, although their whiteness fluctuated slightly. This was similar to the result reported by Shaterikhalilabad and Yazdanshenas (2013b). The mechanism for the chemical reduction of graphene oxide is often regarded as two-step S_N2 nucleophilic reactions followed by one step of thermal elimination. $Na_2S_2O_4$ is an efficient reducing agent owing to its lower electrode potential ($E\ SO_3^{-2}/S_2O_4^{-2} = -1.12$ V) than ascorbic acid and thiourea in alkaline solution (Zhou et al. 2011; Shaterikhalilabad and Yazdanshenas 2013a). And it is ready to dissociate two protons functioning as a

nucleophile. Epoxide and hydroxyl groups of GO molecules are attacked by a nucleophile with a backside S_N2 nucleophile reaction, resulting in the formation of an intermediate and water, and then the intermediate may undergo thermal elimination to produce the reduced GO (Gao et al. 2010; Zhou et al. 2011; Shaterikhalilabad and Yazdanshenas 2013a). Accordingly, $Na_2S_2O_4$ was used as the best reducing agent to examine the effect of process variables on the electronic conductivity of the RGO-dyed cotton fabric. Figure 2b, c shows that the surface resistance and whiteness of the RGO-dyed cotton fabric gradually decreased with an increasing $Na_2S_2O_4$ concentration and reduction temperature, and the decreasing tendency became level at 3.0 % $Na_2S_2O_4$ concentration or at higher than 50 °C. This result indicated that a higher $Na_2S_2O_4$ concentration and temperature enhanced the darker color and the formation of RGO on cotton fabric. A reason is that the electrical conductivity is perhaps the best indicator

Fig. 2 Surface resistance and whiteness of dyed cotton fabric at different reduction conditions

of the extent to which the GO has been reduced to graphene (Khan et al. 2012). When the GO-dyed cotton fabric was reduced for 40 min, the lowest surface resistance and whiteness were achieved. Further increasing the reduction time resulted in a small change in its surface resistance and whiteness.

Characterization of GO-dyed cotton fabric before and after reduction

*FE-SEM observation*

Figure 3a shows the SEM image of the original cotton fiber, which appears as a flat ribbon-like structure with occasional convolutions along its length. Some RGO sheets could be observed on cotton fibers after GO dyeing and reduction, as seen in Fig. 3b, c. A higher magnification (2000×) FE-SEM image of the RGO-dyed cotton (Fig. 3d) show that the wrinkled RGO sheets agglomerated and formed a hierarchical structure, thus increasing the surface roughness, which was similar to the SEM observation by Krishnamoorthy et al. (2012) and Ge et al. (2014).

XPS analysis

According to the survey scan from Fig. 4a, the GO-dyed cotton fabric surface was composed mainly of carbon and oxygen elements (the hydrogen element is not detectable in XPS analysis). The contents of carbon and oxygen were 71.27 and 28.27 %, respectively. After reduction, the content of carbon for the
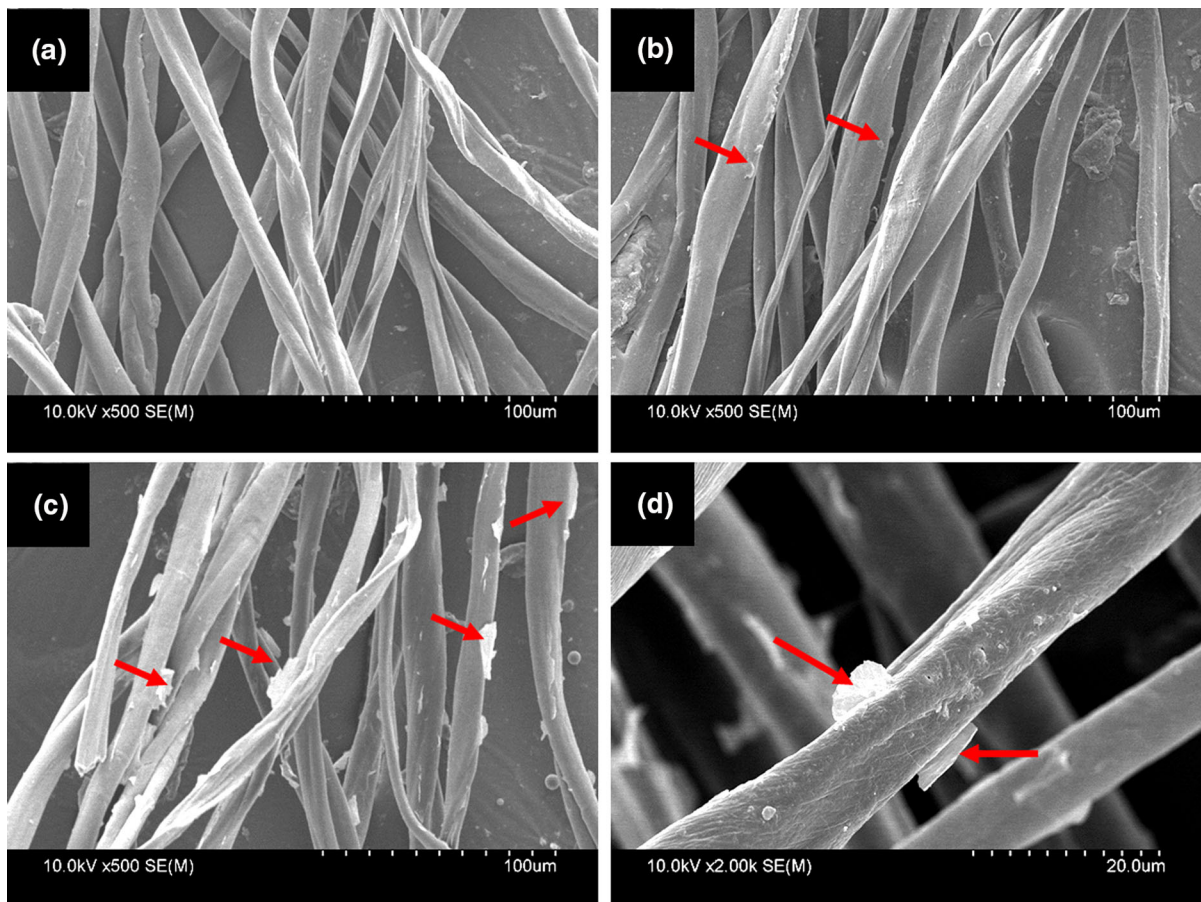
Fig. 3 FE-SEM images of **a** original cotton fibers, **b** RGO-dyed cotton fiber with two dyeing cycles, **c** RGO-dyed cotton fiber with eight dyeing cycles and **d** a higher magnification image of (**c**)
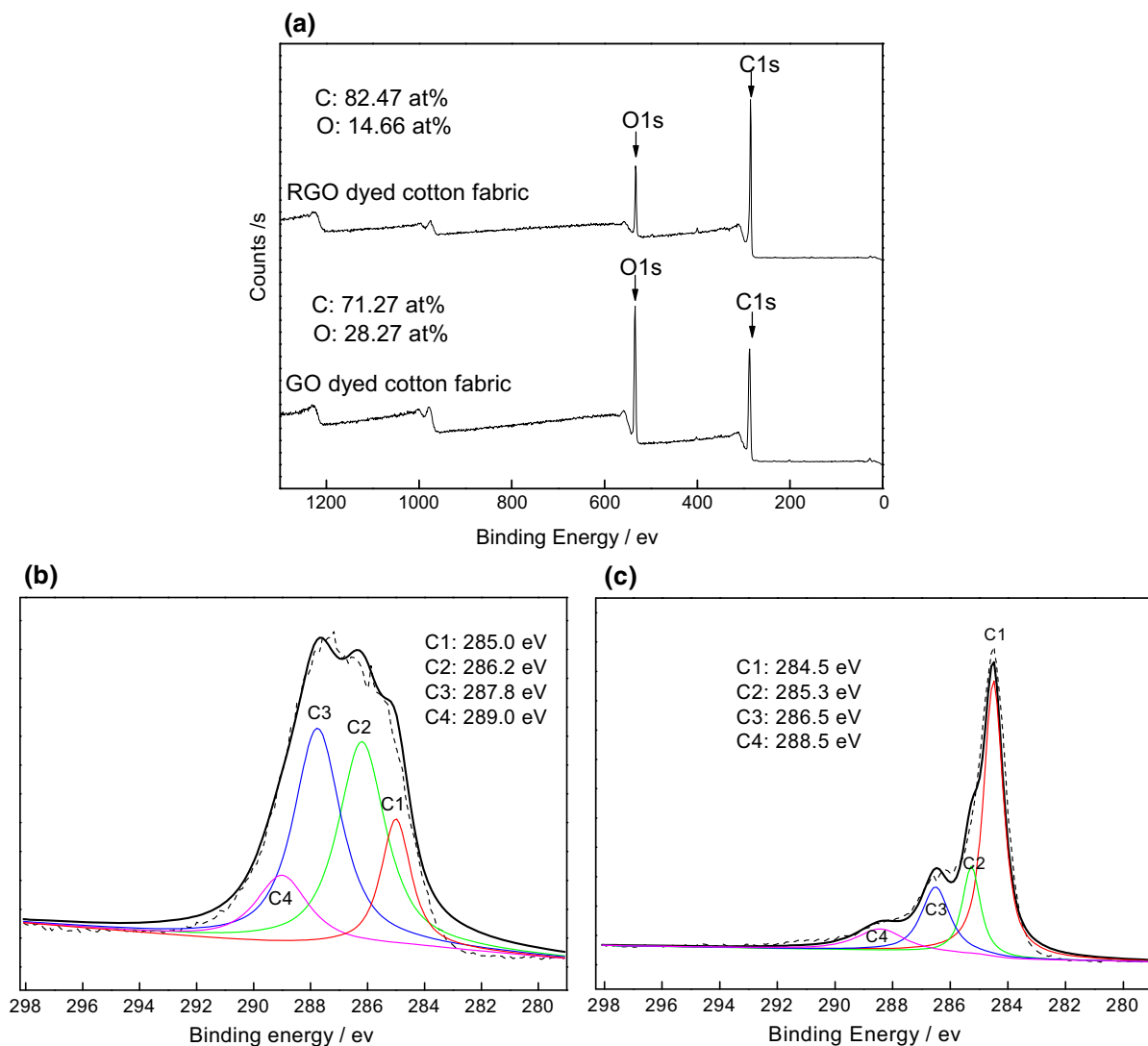
Fig. 4 XPS survey spectra of RGO-dyed cotton fiber (**a**) and high-resolution C1s XPS core level spectra of GO-dyed cotton fiber with four dyeing cycles before (**b**) and after reduction (**c**)

GO-dyed cotton fabric increased to 82.47 %, whereas its oxygen content exhibited a reverse trend. More importantly, the high-resolution carbon C1s spectrum was resolved into four symmetric Gaussian components, C1 (C–C), C2 (C–O), C3 (C=O) and C4 (O–C–O) (Tang et al. 2012; Waltman et al. 1993; Stankovich et al. 2007) (Fig. 4b, c) for the dyed cotton fabric. After reduction, its main peak related to the C–C remains was slightly changed, but the intensity of the three main peaks related to the oxygen group was much smaller. These results are consistent with the

above elemental analysis data and indicated a significant de-oxygenation by the reduction process for GO-dyed cotton fabric.

Raman analysis

Figure 5 shows that the D and G bands of the GO-dyed cotton fabric were centered at 1345 and 1593 cm^{-1}, respectively. After reduction, its Raman spectrum also contained both D and G bands (shifted to 1584 and 1352 cm^{-1}, respectively). However, the ratio of the
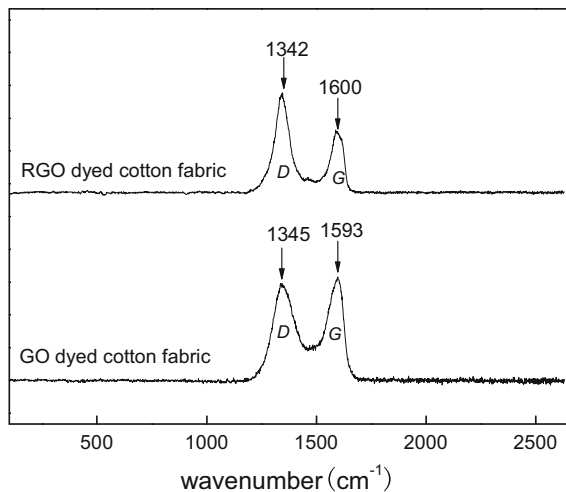
Fig. 5 Raman spectra of RGO-dyed cotton fabric with six cycles before and after reduction

intensities (I_D/I_G) was significantly increased from 0.94 for GO to 1.64 for RGO. This change indicated that some new and small sp2 domains were formed, and the average size of the crystalline RGO increased after reduction (Stankovich et al. 2007; Li et al. 2014).

Effect of dyeing process cycles

A series of GO-dyed cotton fabrics was prepared using different numbers of dyeing processes, and then reduced with Na₂S₂O₄. The electrical resistance of the resulting RGO-dyed cotton fabrics was tested
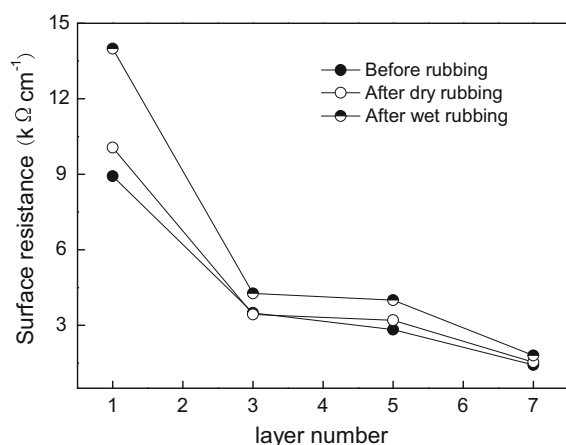

Fig. 6 Surface resistance of RGO-dyed cotton fabric as a function of GO dyeing cycles before and after rubbing

before and after rubbing, and the results are presented in Fig. 6. Furthermore, their EIS measurements were carried out in the $10–10^7$ Hz frequency range, and the results are shown in Fig. 7.

Figure 6 shows that there is a significant decrease of the surface resistance of the RGO-dyed cotton fabrics from the first to the third dyeing process. The surface resistance slightly decreased with further increasing, and the lowest surface resistance value of 1.43 kΩ cm^{-1} was achieved at the seventh dyeing process. On the other hand, Fig. 7 shows that the original cotton fabric has |Z| values higher than 10^8 Ω cm^{-2} for low frequencies. The |Z| values can be observed with varying frequency. When cotton fabric was dyed with GO and successively reduced with Na₂S₂O₄, a |Z| decrease of about six orders of magnitude was obtained (185 Ω cm^2 for 10.0 Hz). It is worth noting that the |Z| value decreases with an increasing number of dyeing processes. When the seventh layer of RGO was deposited on the fabric, the |Z| value lowered to 5.5 Ω cm^2 for 10.0 Hz. According previous works (Molina et al. 2013b; Shaterikhalil-abad and Yazdanshenas 2013a; Fugetsu et al. 2010), the electrical conductivity increases with the increasing graphene content of the substrate, which can be attributed to the improved orientation and structure of the graphene layer. In the first dyeing process, there may be a poor contact between the RGO sheets on the fabric, thus limiting their electrical conduction. Increasing the number of dyeing cycles could add more RGO sheets on the fabric surface to obtain a better RGO sheet-to-sheet connection for constructing an infinite network of connected paths, favoring the electrical conductivity of the fabrics. Besides, Fig. 6 also shows that after dry rubbing, a similar surface resistance value was obtained for all the fabrics dyed with different RGO layers. However, wet rubbing increased the surface resistance, and the difference in surface resistance between the original and dyed fabrics declined as the number of dyeing processes increased. Little change in the surface resistance was found at the seventh dyeing process. These finds revealed that the fabrics with more RGO dyeings have excellent rubbing fastness, indicating better wear resistance of the RGO-dyed cotton fabrics.
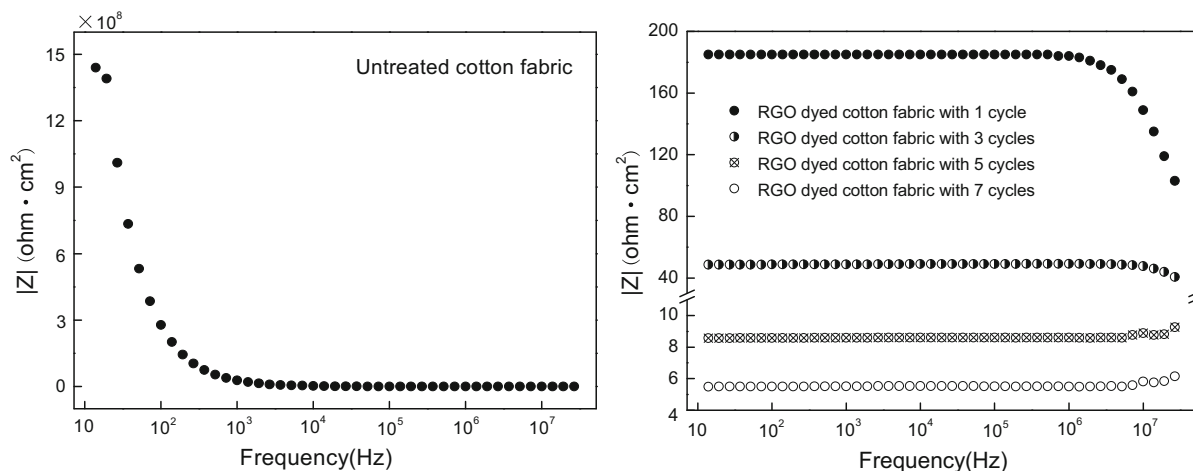
Fig. 7 Dependence of the impedance modulus on AC frequency for dyed cotton fabric with different GO-dyeing cycles

Conclusions

Cotton fabric could be dyed with GO aqueous dispersion using a simple conventional exhaustion dyeing and chemical reducing process to prepare the electrically conductive cotton fabric. SEM, XPS and Raman spectrum analysis confirmed that wrinkled RGO sheets were formed on the surface of cotton fibers after GO dyeing and reduction. Increasing GO concentration or dyeing temperature and time could enhance the GO content on the dyed cotton fabrics and decreased their electrical surface resistance. However, increasing pH increased the surface resistance and whiteness of the dyed cotton fabric because of the enhanced charge repulsion between the GO molecule and cotton fiber. Furthermore, three reducing agents including ascorbic acid, thiourea and $Na_2S_2O_4$ showed a significant reducing effect on the transformation of GO into RGO on cotton fabric. $Na_2S_2O_4$ had a stronger reduction performance than the other two reducing agents at the same conditions. A higher $Na_2S_2O_4$ concentration and elevation of temperature decreased the surface resistance and whiteness of the dyed cotton fabric, thus leading to an increase in its RGO content and electrical conductivity. Little change in its surface resistance was observed when the $Na_2S_2O_4$ concentration was over 3.0 %, especially at higher than 50 °C. The lowest surface resistance and whiteness were obtained when the GO-dyed cotton fabric was reduced with $Na_2S_2O_4$ for 40 min. An increasing number of dyeing cycles resulted in a decrease of the surface resistance and impedance modulus of the RGO-dyed fabric. The lowest surface resistance and impedance modulus were 1.43 kΩ cm^{-1} and 5.5 Ω cm^2 for 10.0 Hz, respectively, at the seventh dyeing cycles. Dry and wet rubbing caused a slight increase in their surface resistance, particularly for the fabric with more RGO dyeings, demonstrating that the obtained RGO layers on the surface of the cotton fabric were very flexible and had better abrasion resistance. To sum up, the accepted electrically conductive cotton fabric was found when it was prepared with a 0.50 % GO aqueous dispersion and 3.0 % $Na_2S_2O_4$ as a reducing agent at the desired temperature and time using a simple exhaustion dyeing process.

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