



## SUSTAINABLE REDUCTION OF GRAPHENE OXIDE ON POLYESTER SPUNBOND NONWOVEN FABRIC USING RED ONION PEEL EXTRACT

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**Abstract:** Graphene oxide (GO) coated conductive textiles have attracted significant attention due to their potential applications in smart textiles, sensors, and electromagnetic shielding materials. However, conventional chemical reduction methods for GO typically involve toxic reducing agents that pose harmful risks to both environmental and human health. Therefore, environmentally friendly reduction strategies have become increasingly important. In this study, graphene oxide was coated onto polyester spunbond nonwoven fabrics using dip-coating and blade coating methods. Subsequently, a green reduction approach was applied using the methanol extract of red onion peel as a natural reducing agent. The morphology, chemical structure, thermal stability, and electrical properties of the obtained samples were characterized by scanning electron microscopy (SEM), Raman spectroscopy, thermogravimetric analysis (TGA), and surface electrical resistance measurements. The results demonstrate that agricultural waste-derived natural extracts can serve as effective and sustainable reducing agents for the preparation of conductive graphene-based textile structures. This environmentally friendly approach offers a promising route for the development of conductive nonwoven materials for advanced functional textile applications.

**Key words:** graphene oxide, green reduction, waste management, nonwoven fabric, electrical conductivity.

### 1. INTRODUCTION

One of the most significant global issues today is the valorization of waste. The rapid increase in population and imbalances in supply chains have led to a growing global concern regarding food waste. Cultivating plants requires intensive labor and significant economic resources. Therefore, using only the edible parts while discarding the rest leads not only to food waste but also to a loss of labor and resources. From a sustainability perspective, the utilization of waste materials has become increasingly important. About 20% of food waste occurs during production, 1% during processing, 19% during distribution, and 60% at the consumer and household level. Fruit and vegetable residues constitute a major portion of this waste, accounting for nearly 42% of the total. Although some food industry waste is currently composted or used as animal feed and fertilizer, recovering valuable bioactive compounds from these wastes and reusing them as sustainable resources offers a more effective waste management approach. Numerous studies have demonstrated that fruit and vegetable peels contain significant amounts of bioactive compounds. Furthermore, previous research has reported that the bioactive content of fruit and vegetable peels and seeds may be even higher than that of their edible tissues [1-3]. For the reduction of graphene oxide (GO), the chemical reduction method—an effective and relatively simple procedure—is commonly applied. Through this process, oxygen-containing functional groups are removed from the GO structure [4]. Conventional chemical reduction methods commonly use toxic reducing agents such as hydrazine hydrate, dimethylhydrazine, sodium borohydride, and hydroquinone, which are harmful to both human health and the environment [5]. Moreover, hazardous by-products formed during these reactions can attach to the reduced graphene oxide structure and limit its potential



applications. For this reason, environmentally friendly reduction methods have attracted increasing attention [6, 7]. In green reduction studies, natural extracts obtained from different plant parts, including leaves, fruits, roots, seeds, and whole plants, are widely used [8]. Studies have shown that red onions contain bioactive compounds such as antioxidants. Red onion peel is rich in phenolic compounds, mainly flavonoids, which are higher than those of the edible parts [9, 10]. Therefore, in this study, the potential of red onion peel extract on reducing oxygen-containing functional groups in GO, which is coated on a nonwoven textile substrate for applications in electro-conductive functional textiles.

## 2. MATERIALS AND METHOD

### 2.1 Materials

Graphite flakes were purchased from Sigma-Aldrich. Hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 35%), sulfuric acid ( $\text{H}_2\text{SO}_4$ , 95%–98%), phosphoric acid ( $\text{H}_3\text{PO}_4$ ), potassium permanganate ( $\text{KMnO}_4$ ), boric acid ( $\text{H}_3\text{BO}_3$ ), and hydrochloric acid ( $\text{HCl}$ , 37%) were purchased from Merck. All chemicals were of analytical reagent grade and used without further purification. Distilled water was used throughout the experiments. Polyester spunbond nonwoven (NW) fabric with a mass per unit area of  $25 \text{ g/m}^2$  was used as a substrate. Red onion peels (ROP) were obtained from the local market and used as a reducing agent to reduce the graphene oxide.

### 2.2 Synthesis of Graphene Oxide

Graphene oxide was synthesized using the improved Hummers' method using flake graphite. Briefly, 360:40 mL of a mixture of  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$  was placed into a flask, to which 3g of graphite flakes were added. The flask was gradually filled with 18g of  $\text{KMnO}_4$ . Following the full dissolution of the  $\text{KMnO}_4$ , the reaction was placed in an oil bath, heated to  $50^\circ\text{C}$ , and stirred for 12 hours. The reaction was then poured onto 400 mL of ice after being cooled to room temperature. 6 mL of 35%  $\text{H}_2\text{O}_2$  was added to terminate the reaction. The suspension was purified using ethanol (2 $\times$ ) and 1 M  $\text{HCl}$  by centrifugation. After washing, the centrifugation process was maintained until a pH of 4-5 was reached. The resulting sample was dried at  $60^\circ\text{C}$  to obtain solid GO nanosheets [11].

### 2.3 Ultrasonic Extraction

The red onion peels were removed, washed with distilled water, and dried in an oven at  $70^\circ\text{C}$ . The dried peels were ground into powder. 30 g of red onion peel powder was extracted with the mixture of 100 mL (1:1) methanol: distilled water by using the bath sonication at  $50^\circ\text{C}$  for 60 min. After the extraction, the mixture was filtered and then centrifuged for 10 min at 6000 rpm. The supernatant was stored at  $4^\circ\text{C}$  and used as a reducing medium.

### 2.4 GO Coating and Reduction Process of Polyester Spunbond Nonwoven Fabric

The polyester spunbond nonwoven fabric was first dip-coated with the 2 g/L aqueous dispersion of GO. The aqueous dispersion of GO was prepared by adding 2 g of GO into 1 L of distilled water, placing it into the ultrasonic bath for an hour at room temperature, and then transferring it to the magnetic stirrer to obtain a homogenous dispersion. The polyester nonwoven fabric was dip-coated with the as-prepared GO dispersion for about 10 min at ambient conditions (The sample coded as D-GO-NW). Secondly, the GO-coated polyester nonwoven fabric was blade-coated with 25 g/L of GO paste (The sample coded as B-GO-NW). The blade-coating paste was prepared with GO and distilled water without any auxiliary chemicals. The blade-coating parameters were fixed as given: blade-coating speed 1 and 2 m/min, blade-fabric distance 35 mm, curing temperature  $190^\circ\text{C}$ , curing duration 180 sec.

The GO-coated polyester nonwoven fabric samples were put into the methanolic extract of red onion peel, and the reaction was heated to  $95^\circ\text{C}$  and held for 24 h. The fabric samples were then rinsed with distilled water to remove the excess reducing agent and dried in an oven at  $95^\circ\text{C}$ . The samples after the reduction were coded as ROP-RGO-NW-1 and ROP-RGO-NW-2, indicating that the blade-coating speeds were 1 and 2 m/min, respectively.

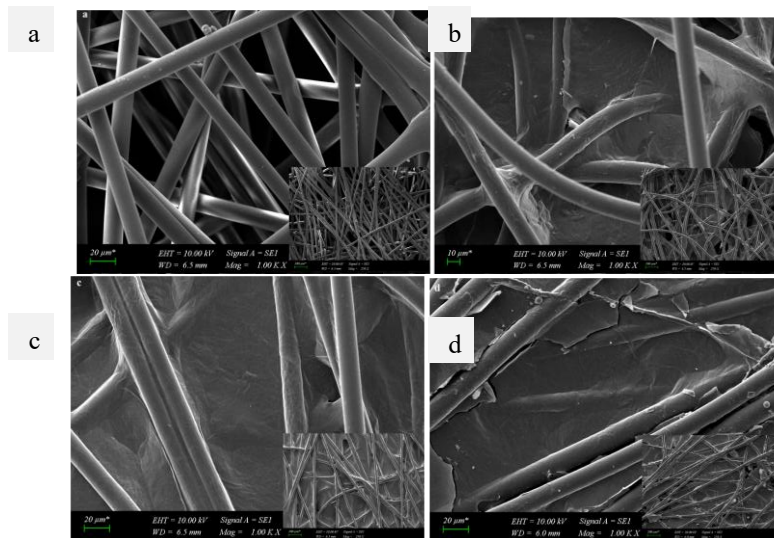
## 2.5 Characterization

Scanning electron microscopy (SEM, Zeiss Evo MA10) was employed to observe the morphology of uncoated and coated samples. The surface of the samples was coated with Au/Pd (Quarum, SC7620) before analysis. Thermogravimetric analysis (Perkin ELMER TGA 8000) was applied to the nonwoven fabric samples at a rate of 10°C/min under nitrogen flow from 25 to 600°C. The Raman spectra were acquired using a WITec Alpha300 RA with a 532 nm laser (WITech, Germany). The electrical resistance of the nonwoven fabric samples was determined using a four-point probe technique. The configuration comprises a sourcemeter (Keithley 2450) and a four-point probe station (Everbeing International Corp.).

## 3. RESULTS AND DISCUSSIONS

### 3.1. Morphological Analysis

Scanning electron microscopy (SEM) images of the polyester spunbond nonwoven fabric, GO dip- and blade-coated, and red onion peel extract-reduced sample are presented in Figure 1. Figure 1a shows the surface of the polyester spunbond nonwoven fabric, where polyester filaments exhibited a smooth and uniform structure. After the GO dip-coating process, GO nanosheets were observed on the fabric surface and between the filaments (Figure 1b). Following the blade-coating application, the fabric surface became more densely covered with GO nanosheets. As shown in Figure 1c, no voids were observed between the polyester filaments, and the filament surfaces were completely coated with GO nanosheets exhibiting the characteristic wrinkled and crumpled morphology of GO. After the reduction process using the methanol extract of red onion peel, cracks appeared on the coated surface (Figure 1d). This observation indicated that the oxygen-containing functional groups in the GO structure were removed during the reduction reaction, leading to the formation of a more rigid and compact reduced GO structure.



**Fig.1.** SEM images at 1000× magnification of NW (a), D-GO-NW (b), B-GO-NW (c), and ROP-RGO-NW-1 (d)(inside images are at 250× magnification).

### 3.2. Thermal Gravimetric Analysis (TGA)

The thermal behavior of the samples was analyzed using a thermogravimetric analysis (TGA) instrument. As observed from the TGA curves presented in Figure 2, all samples exhibited similar thermal degradation behavior. The thermal degradation process occurred in two stages. In the first stage, a slight mass loss was observed up to 100 °C, which was attributed to the removal of adsorbed water molecules from the samples [11]. In the second stage, a significant mass loss occurred within the temperature range of 380–480 °C. This could be explained by the chain scission of ester bonds and the

formation of vinyl ester and carboxylic acid groups. With increasing temperature, the ester bonds continued to break, leading to the formation of volatile low-molecular-weight segments. The release of these segments resulted in a substantial mass loss [12]. The TGA curve indicated that the thermal stability of the red onion peel extract-reduced sample showed a slight improvement. The residual mass percentages at 600 °C were determined as 9.50%, 9.70%, and 12.43% for samples coded NW, D-GO-NW, and ROP-RGO-NW-1, respectively. These results suggested that the reduction process with red onion peel extract enhanced the thermal stability of the sample.

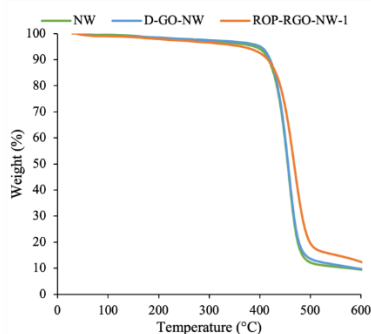


Fig.2. TGA graph of NW, D-GO-NW, and ROP-RGO-NW-1.

### 3.3. RAMAN Analysis

The Raman spectra of the polyester spunbond nonwoven fabric, GO dip-coated sample, and red onion peel extract reduced sample were presented in Figure 3. The polyester nonwoven fabric exhibited characteristic peaks at 1293  $\text{cm}^{-1}$  (C–O), 1616  $\text{cm}^{-1}$  (aromatic ring stretching), and 1728  $\text{cm}^{-1}$  (C=O). No distinct new peak formation was observed in the polyester nonwoven fabric coated with GO via the dip-coating method. This could be attributed to the relatively low amount of GO present on the fabric surface. Following the reduction reaction using red onion peel extract, the Raman spectra of the samples revealed the characteristic D and G bands of carbon-based materials. The D band arises from the presence of defects and impurities in the basal plane of the graphitic structure [13]. The D band was observed at 1364  $\text{cm}^{-1}$ . The G band is associated with the defects related to  $\text{sp}^2$  hybridization in the ordered graphene structure [14]. The G band was observed at 1596  $\text{cm}^{-1}$ . The  $I_D/I_G$  ratio was determined as 0.99. The  $I_D/I_G$  ratio indicated the reduction in the size of planar  $\text{sp}^2$  domains during the reduction of GO and was considered an indicator of structural disorder [13, 15].

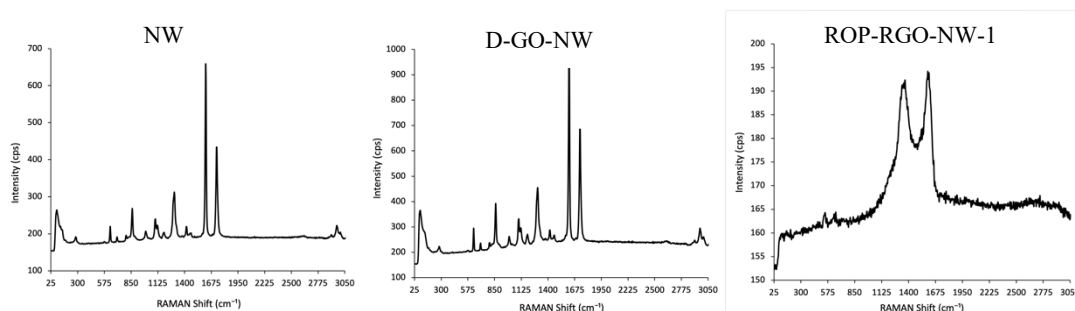


Fig.3. RAMAN spectra of NW, D-GO-NW, and ROP-RGO-NW-1.

### 3.4 Electrical Resistivity

The electrical surface resistance measurement results were presented in Table 1. Due to its inherently insulating nature, the polyester nonwoven fabric exhibited a very high electrical resistance value of  $2.37 \times 10^8$  k $\Omega$ . The GO dip-coated nonwoven fabric sample also exhibited a high electrical resistance due to the insulating nature of GO nanosheets, which arises from the presence of oxygen-containing functional groups [11]. A significant decrease in electrical resistance value was observed after the reduction of GO. The lowest value was obtained in the ROP-RGO-NW-2 sample as  $1.75 \times 10^2$

k $\Omega$ .

The electrical conductivity of the produced samples was also demonstrated by the illumination of an LED bulb in a simple circuit setup. The images presented in Figure 4 demonstrate that the sample reduced using red onion peel extract exhibited sufficient electrical conductivity to power an LED lamp.

*Table 1: The electrical resistivity of samples.*

<i>Sample</i>	<i>Electrical Resistivity (k<math>\Omega</math>)</i>
NW	2.37E+08
D-GO-NW	3.94E+07
ROP-RGO-NW-1	3.08E+03
ROP-RGO-NW-2	1.75E+02



*Fig.4. The digital photographs showing the gloves (a, b), NW (c), D-GO-NW (d), and B-GO-NW (e) do not light the LED, while the ROP-RGO-NW-2 sample lights the LED.*

#### 4. CONCLUSIONS

In this study, graphene oxide (GO) is successfully coated onto polyester spunbond nonwoven fabrics using dip-coating and blade coating methods, followed by a green reduction process using natural extract obtained from red onion peel. The study aims to develop an environmentally friendly approach for producing conductive textile structures while utilizing agricultural waste materials as sustainable reducing agents. SEM analysis confirms that GO nanosheets are successfully deposited on the polyester filament surfaces and within the inter-fiber spaces of the nonwoven structure. Raman spectroscopy results support the successful reduction of GO. Thermogravimetric analysis demonstrates that the reduced sample exhibited slightly improved thermal stability compared to the untreated and GO-coated fabrics. Electrical surface resistance measurements show that the reduction process significantly enhanced the electrical conductivity of the samples. The lowest electrical resistance value (174.75 k $\Omega$ ) is obtained. Furthermore, the electrical conductivity of the obtained sample is visually demonstrated through the illumination of an LED lamp in a simple circuit setup.

Overall, the results indicate that plant-based waste extracts can be effectively used as green reducing agents for the preparation of conductive graphene-based textile materials. This sustainable approach not only contributes to the valorization of agricultural waste but also provides an environmentally friendly



alternative to conventional toxic reducing agents for the production of functional conductive nonwoven textiles.

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