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Short communication

Continuous dyeing of graphene on cotton fabric: Binder-free approach for electromagnetic shielding

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ABSTRACT

In this paper, Graphene oxide dyed on the cotton fabric through an efficient and industrially used continuous dyeing method. The graphene oxide dyed cotton fabric was reduced by using green reducing L-ascorbic at 100 °C microwave-assisted reduction. The effect of with different weight percent of rGO on sheet resistance and active electromagnetic interference (EMI) shielding of the studied. The surface modification of the fabric was achieved by using dopamine; the results showed significant improvements for the adhesion of (rGO) distributed uniformly on the fibre surface. Also, the rGO dyed fabric showed higher electrical conductivity 2.3×10^{-1}S/cm^{-1} and EMI SE of −26 to −35 dB in the range of 30–1530 MHz in X-band.

1. Introduction

The electromagnetic waves are highly dangerous public pollution after noise, water and air during the last several decades which have an adverse effect on human health causing several diseases such as cancer, tumour, Alzheimer's, and Parkinson's [1–3]. In this concern, several studies have made by using different inorganic materials such as silver, copper, gold, zinc, titanium, and aluminum-coated on textiles, composites, and yarns [4–7]. The use of the organic compounds is an alternative approach for the (EMI) shielding, such as CNT, carbon black, graphene, graphene oxide, rGO and PEDOT due to their higher electrical conductivity [8–10]. Furthermore, these organic compounds may also be combined with conductive polymers to enhance the electrical conductivity and adhesion properties as compared to common inorganic compounds [11–13,22]. Graphene is a 2D material with the electrical conductivity of ~6000 Scm^{-1} [14], where the conductivity required for the EMI shielding is nearly 10^{-3} Scm^{-1}, which proves that graphene is a highly promising material for future application of EMI shielding and electrical conductivity of textile-based materials. Some other techniques such, layer by layer, vacuum filtration, brush coating, direct electrochemical deposition, electrophoreses and screen printing have been reported for the fabrication of textiles with (GO) and (rGO) [15–17]. Therefore in this research, we approached the most efficient technique for mass production of rGO coating as a continuous dyeing process (1–20 cycles) on textiles for EMI shielding.

2. Experimental

2.1. Materials

Plain weave 100% cotton fabric (white color) was collected from Zhejiang Sci-Tech University fabric lab. Multilayer graphite powder (purity > 99.95%) metal basis was purchased from Aladdin industrial corporation. All the other chemicals such as H_2SO_4, HCl, H_2O, $KMnO_4$ have been used as analytical grade without further purification.

2.2. Synthesis GO

GO was synthesized using modified Hummer's method, as reported in our previous study [18].

2.3. Continuous dyeing process

Briefly, the graphene oxide dispersion in distilled water was dissolved with (1.0 g/L^{-1} in 100 mL) and improved the dispersion using ultrasonication process for an hour. The GO dye solution was used for the continuous dyeing of cotton fabric using binder-free approach and continuously reduced using a green, reducing agent (L-ascorbic acid) at 100 °C for 5 min, and curing was achieved at 120 °C for 2 min. The resultant samples were defined as rGO-1, 2, 3, 4, 5 for 1, 5, 10, 15, 20 dyeing cycles respectively.

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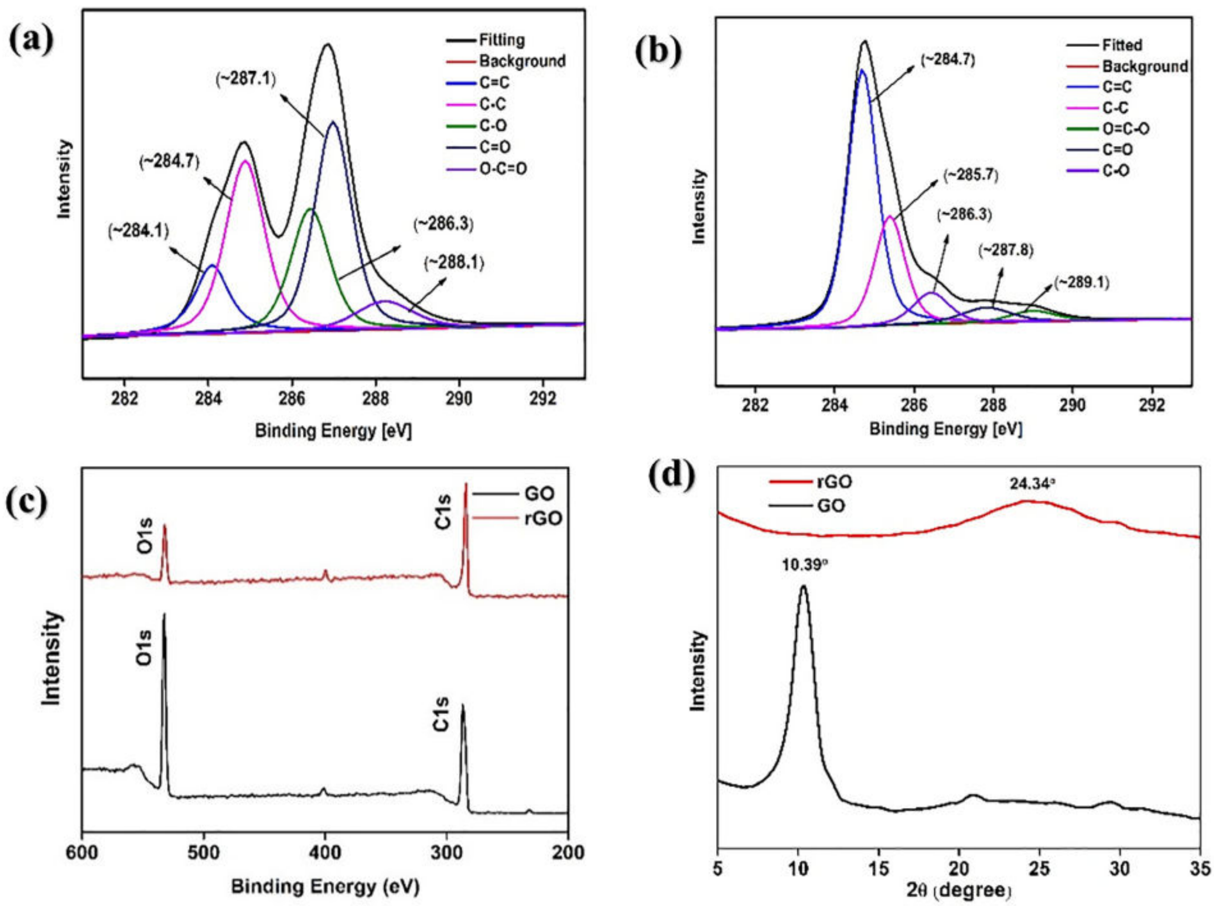

Fig. 1. XPS Analysis of GO and rGO (a) O1s, (b) C1s, (c) Survey peaks, (d) XRD analysis of GO and rGO.

3. Results and discussion

3.1. Elemental studies

The C1s XPS spectra of GO shows convoluted five peaks located at (~284.1 eV, ~284.7 eV, ~286.3 eV, ~287.1 eV, and ~288.1 eV) binding energy. These peaks are assigned to electrochemical structures of (C═C, C─C, C─O, C═O and, O─C═O) for the carbon atoms. Whereas, the O─H Peaks are located with broader peak intensity, which is reduced when GO is converted into rGO as shown in the Fig. 1a and b respectively [19]. The intensity of the C1s peak for rGO dyed cotton fabric positioned at 286.6 eV and 288.0 assigned to (C─C) bonds located at 284.0 eV with strong bonding. The carbon content (~69.8% to ~87.8%) with C/O ratio of 7.2 was observed, which may be attributed to the significant reduction of GO into rGO. Furthermore, the C/O ratio has been improved from 2.3 to 7.2 with increasing the dyeing cycles of the cotton fabric, as shown in the (Table 1).

The XRD diffraction for the GO patterns exhibited reflection peak at 10.39° of 2θ associated with the (001) plane, with a spacing of 0.85 Å for the GO dyed fabric. However, after chemically and thermally reduction of GO to rGO, the XRD diffraction pattern shows a broad peak with a maximum at 24.34°, corresponding to a turbostratic structure with a mean spacing of 3.6 Å.

Table 1
C/O ratio from wide-angle scan XPS analysis.

Sample	C (%)	O (%)	C/O
Treated cotton	69.8	30.2	2.3
rGO-1	77.1	22.9	3.3
rGO-2	81.6	18.4	4.4
rGO-3	84.7	15.3	5.5
rGO-4	86.3	13.7	6.3
rGO-5	87.8	12.2	7.2

3.2. Raman spectroscopy analysis

Raman spectroscopy provides the information based on the inelastic (Raman) scattering of a molecule illuminated by monochromatic light. I_D/I_G ratio for GO coated fabric was determined to be 0.92 (Fig. 2). After the reduction, (Fig. 2) showed that the I_D/I_G ratio for rGO coated cotton fabric (rGO-1 to rGO-5) was increased 1.17 and 1.41 respectively due to the restoration of sp2 carbon and decreased in the average sizes of sp2 domains upon reduction. Increasing of dyeing cycle increased the loading of rGO or content percent. We achieve better reduction for repetitive reduction & curing process, which obtained a higher I_D/I_G ratio after 20 dyeing cycle. The higher intensity in D band also presents, that the more isolated graphene domains were present in RGO as compared to GO and also due to the removal of oxygen moieties from GO after reduction.

3.3. Morphological study

The FESEM image (Fig. 3a, b and c) presents the higher binding on the fibre surface which may attribute the process approach in the study for better use of EMI-shielding efficiently, which is attributed to the higher loading of rGO and covering the porosity of the inter-fibre spacing in the weave structure and fibre to fibre spacing respectively. Furthermore, the SEM images were further analyzed using Mountain maps software v.7.0 (https://www.digitalsurf.com) using SEM colour rendering module, as shown in Fig. 3a´, b´ and c´ respectively.

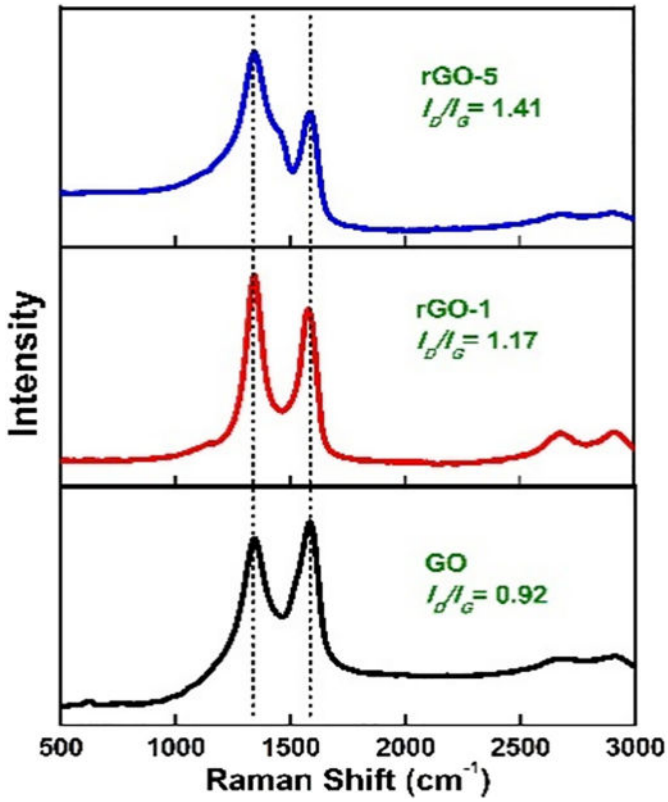

Fig. 2. Raman spectra of GO and rGO coated cotton fabric.

Moreover, thickness of the rGO layer was estimated by a surface profilometer according to the ASTM E2338–17 standards from the five different places of the both control and experimental samples [20]. It was found that the thickness of the rGO layer on the cotton fabric was ranging from 80 to 374 μm for rGO-1~rGO-5.

3.4. Tensile properties

The fabrication of graphene-based coating of rGO on textile substrate showed significant improvements in tensile strength. Whereas, the cellulose fibres are susceptible due to the green reducing agents, at temperature 120–140 °C. Whereas, the tensile strength of the fabric as shown in (Fig. 3a) increased from 25 MPa~42 MPa, which may attribute the sufficient coating layer of rGO on the surface of fibres, revealed in SEM images Fig. 2. On the other hand, the strain% (Fig. 4a) of the GO dyed fabric was consistently reduced as the dyeing cycle of rGO was increased, due to the strong adhesion of fibre to fibre and even in the weave structure on an individual level. The tensile strength of the rGO dyed cotton fabrics increased 27–30% after five dyeing cycles, which further improved up to 60–68% after 15–20 number of dyeing cycles that is the first time reported in this research.

3.5. Electrical conductivity and EMI shielding

The conductivity of the rGO dyed fabrics was measured from Keysight electrometer (model 34420A) at room temperature. The conductivity ‘σ’ obtained from the following Eq. (1).

$$\sigma = \frac{1}{\rho} = \frac{l}{Ra} \tag{1}$$

where ‘ρ’ is the electrical resistivity, ‘*l*’ is the thickness, ‘a’ is the cross-section area, and ‘R’ is the electrical resistance of the samples.

The EMI Shielding effectiveness (EMI SE) was measured by network analyzer (model FY800), supplied by Wenzhou Fangyuan Instrument Company Limited. The EMI Shielding effectiveness describes the ability to reduce the electromagnetic wave ratio of incident power (P_I) to the transmitted power (P_T) in decibel by S-parameters, S_{11} and S_{21} from Eqs. (2)–(4) [21].

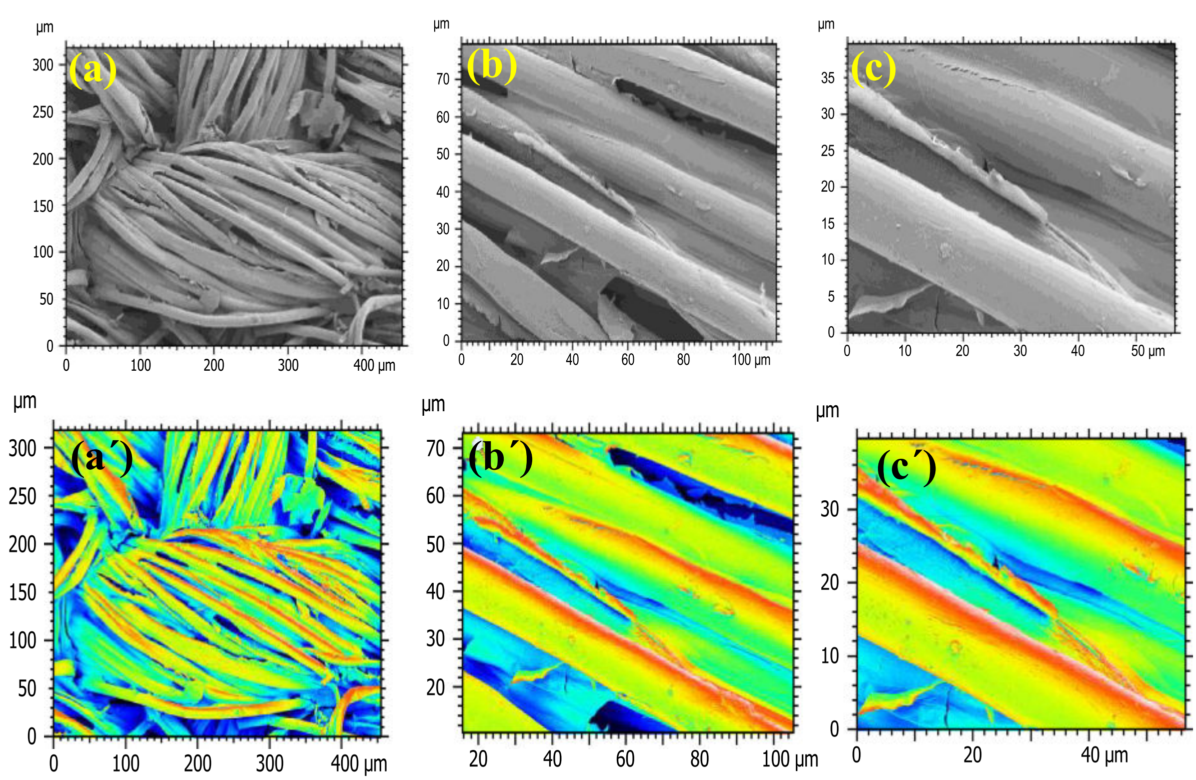

Fig. 3. (a), (b) and (c) FESEM micrographs of rGO dyed cotton fabric at different magnification and (a´), (b´) and (c´) colour rendering image of respective SEM images.

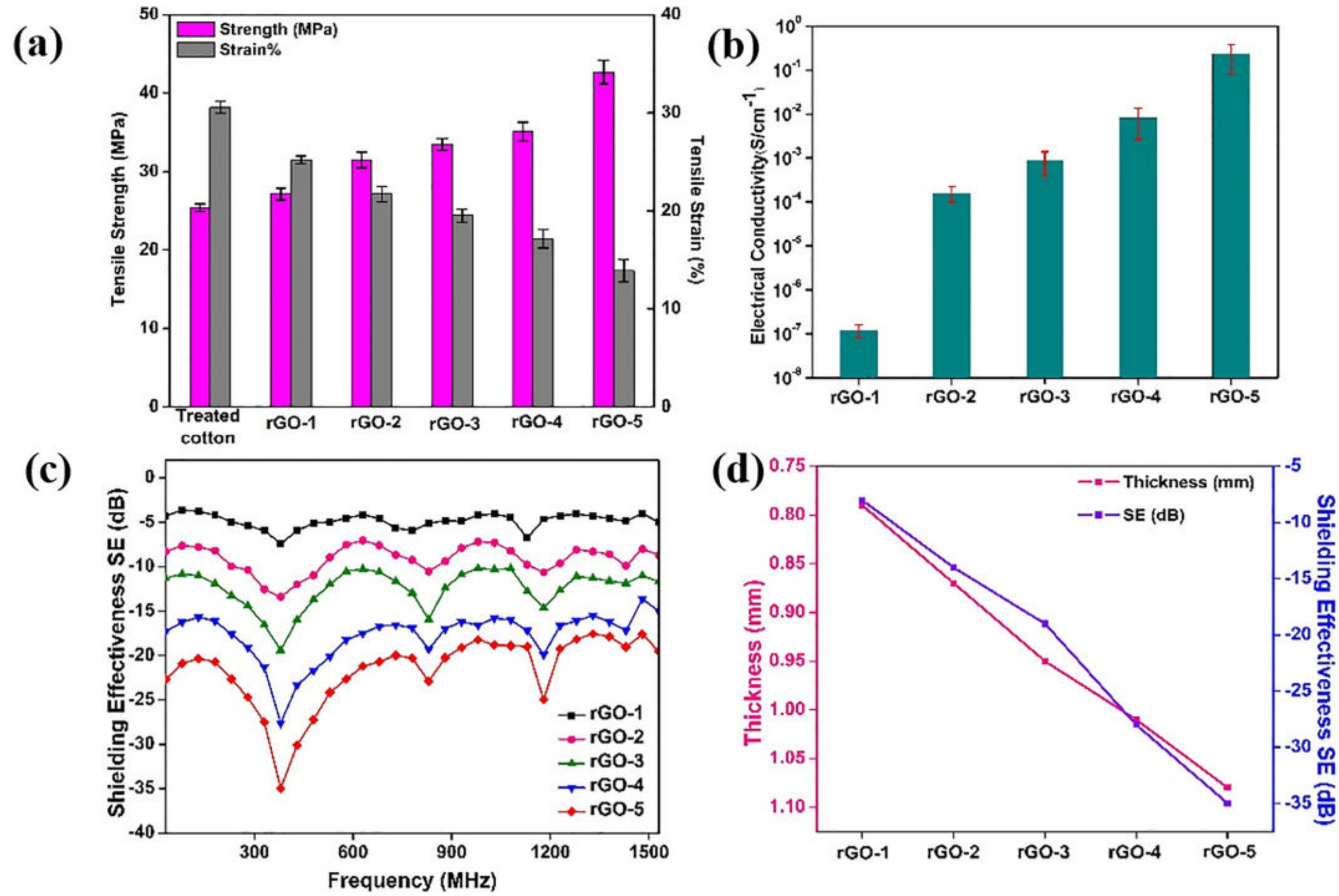

Fig. 4. (a) Tensile strength and strain%, (b) Electrical conductivity, (c) EMI Shielding effectiveness of rGO dyed cotton fabric, (d) Thickness on maximum shielding effectiveness.

$$SE_T = 10\,log\,(P_I/P_T) \tag{2}$$

$$|S_{11}| = \sqrt{P_R/P_I} \tag{3}$$

$$|S_{21}| = \sqrt{P_T/P_I} \tag{4}$$

where P_R refers to the power of the reflected wave. The reflection coefficient (R), transmission coefficient (T), and absorption coefficient (A), can be calculated using Eqs. (5)–(8).

$$R = |S_{11}|2 \tag{5}$$

$$T = |S_{21}|2 \tag{6}$$

$$A = 1 - R - T \tag{7}$$

The *SE* values contributed by reflection (SE_R), absorption (SE_A), and the total $SE(SE_T)$, of materials, are determined by

$$SE_R\ (dB) = 10\log(1 - R) \tag{8}$$

$$SE_A\ (dB) = 10\log\,[T/(1 - R)] \tag{9}$$

$$SE_T\ (dB) = SE_R + SE_A \tag{10}$$

where −20 dB to −25 dB describes that the EMI shielding of 99% and 99.99% with the incident (P_I) electromagnetic wave shielding [1]. The (EMI SE) was measured of dopamine treated cotton fabric, which has almost have no shielding effect. The result showed that the sample (rGO-1) has shielding efficiency around −8 dB, which resultant after rGO dyed on fabric the electrical conductivity and towards improved EMI SE performance. After 20 dyeing cycles, the content percent of rGO and layers significantly increased the conductivity form 1.2×10^{-7} S/cm^{-1} to 2.3×10^{-1} S/cm^{-1} as well as EMI SE improved to −35 dB on 30–1530 MHz with X band as shown (Fig. 4b and c). Whereas, the thickness of the fabric was increased only 1.08 mm (rGO-5) (Fig. 4d). It is clear from the results that, the increase of rGO layers during dyeing cycles improved the conductivity and reduced the porosity of the fabric; as a result, the EMI SE effectiveness was enhanced significantly.

The SE_A and SE_R were analyzed to identify the predominant EMI shielding mechanism for the rGO coated cotton fabric. It was found that SE_R contributes more than ~75% of the total SE_T for all rGO coated cotton fabric studied (Fig. 5a & b), indicating that the attenuation of electromagnetic wave energies into thermal/internal energies was dominant. However, the contribution by SE_A to the total SE_T was minimal.

This research presents the excellent result for electromagnetic wave protected rGO dyed cotton fabric without the addition of binder or metallic compounds, which can be develop wearable textiles for daily uses.

4. Conclusions

In brief, the surface electrical conductivity and EMI shielding performance regarding to the SE of rGO dyed cotton fabric were investigated in this study. The overall deposition rate along with the higher electrical performance of the fabric in terms of sheet resistance and shielding effectiveness of the rGO dyed fabric with a few dyeing cycles, which attributed the higher content% of rGO to enhance the SE in the resultant fabrics. Finally, it was concluded from the experimental results that, the overall adhesion and loading of rGO was increased after treatment and modification of cotton fabric using dopamine as compared to natural fabric. In the future, the research would be carried out

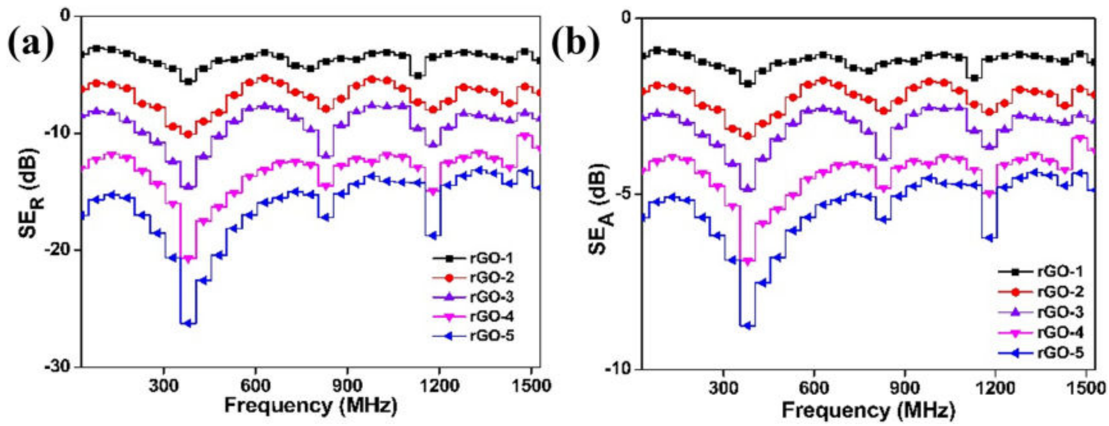

Fig. 5. EMI Shielding refection & absorption of rGO dyed cotton fabric (a) SE$_R$ and (b)SE$_A$.

for the design and development of wearable smart textiles.

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