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Electrically conductive, hydrophobic, UV protective and lightweight cotton spunlace nonwoven fabric coated with reduced graphene oxide

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Abstract: In this study, cotton spunlace nonwoven fabrics with four different basis weights were coated with graphene oxide (GO) by conventional dip coating method which is one of the most simple and effortless process in textile industry. The graphene oxide coated cotton nonwoven fabrics were immersed in an aqueous sodium dithionite solution in order to obtain reduced graphene oxide (RGO) coated cotton spunlace nonwoven fabrics. The obtained nonwoven fabrics became electrically conductive with very low surface electrical resistivity of 5.98×10^2 W/sq for the 70 g/m² basis weight nonwoven fabric. The color measurements and reflectance spectrophotometry were performed in order to identify the coating and reduction process of GO on cotton nonwoven fabrics. The hydrophobic characteristic of the GO and RGO coated cotton nonwoven fabrics were determined by the means of water contact angle. The ultraviolet (UV) blocking ability of the cotton nonwoven fabric both coated with GO and RGO were analyzed by the UV transmittance analyzer. The water contact angle results revealed that the hydrophilic cotton nonwoven fabric became hydrophobic due to the reduction of GO. It was also found that the ultraviolet protection factor (UPF) depends on the basis weight of the cotton nonwoven fabric and the reduction of GO.

Key words: Cotton, nonwoven fabric, graphene oxide, coating, reduction

1. Introduction

Functional textile materials have attracted great attention for their electrical conductivity [1, 2], antibacterial [3, 4], fire retardant [5, 6], superhydrophobic [7, 8], ultraviolet (UV) blocking [9, 10], energy storage [11], sensing properties [12, 13]. These textiles have potential applications in biomedical monitors, high performance textiles, sportswear, wearable displays, military garments [14, 15]. Functional textiles can be prepared by approaches such as spinning, knitting, weaving, coating, and printing. Cotton is the predominant natural fiber in the textile industry because of its natural softness, high hygroscopicity, superior wear comfort and skin-friendliness [16]. Nonwoven fabrics are one of the fastest growing part of textiles of the global textile industry [17]. The nonwoven fabrics have large number of applications such as sound absorption [18], reinforcing fibers [19], healthcare [20, 21], filter media [22], electromagnetic shielding [23, 24] and membrane [25], etc. Therefore, nonwoven fabric can be an ideal supporting material for functional textiles, in particular for electrically conductive textiles. Spunlace nonwoven fabrics are manufactured through entangling loose fiber webs by using jets of water. High velocity jets of water are passed through a closely spaced nozzle to mechanically interlock fibers through displacement, twisting and rearrangement or reorientation to create fabrics [26]. This process provides unique nonwoven fabrics without damaging fibers and without the need for a binder [27]. In the manufacturing process of spunlace nonwoven fabrics, short staple fibers with an average fiber length of 38–40 mm are used [28]. Cotton nonwoven fabric possess a hierarchical structure with high porosity, large surface area and hydrophilic functional groups [14] and also, have lots of advantages such as low cost, light weight, environmentally friendliness [29].

Graphene, one of the allotropes (carbon nanotube, fullerene, diamond) of elemental carbon, is a planar monolayer of carbon atoms arranged into a 2-dimensional (2D) honeycomb lattice [30, 31]. Graphene has attracted great attention because of its excellent properties, such as high thermal and electrical conductivity [32]. Chemical conversion of graphite to graphene oxide (GO) that is a process with low cost and high reaction yield, has emerged to be a viable route to obtain graphene or reduced graphene oxide [30, 33]. Compared to pristine graphite, GO is heavily oxygenated hydroxyl and epoxy groups on the basal plane, in addition to carbonyl and carboxyl groups located at the sheet edges [30, 34]. Hence, GO is highly hydrophilic and readily exfoliated in water, yielding stable dispersion. On the other hand, GO is electrically

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insulating due to the disruption of the conjugated electronic structure by these functional groups. Chemical reduction of graphene oxide usually takes place to obtain electrically conductive form [35, 36]. GO is more suitable for functional textiles because it can be applied to textiles in various techniques, including dip coating [37], screen printing [38], ink-jet printing [39], dyeing [40]. Regarding these various techniques to produce functional textiles, the most widely used method is the dip coating which is simple, easy to apply and scalable [41]. Recently, several studies were reported on the modification of textiles by coating with graphene oxide for obtaining functional textiles. Mengal et al. [42] produced a stable textile electrode from plain woven lyocell fabric by coating with reduced graphene oxide (RGO). Xu et al. [43] coated the cotton fabric with graphene oxide followed by the reduction process in order to obtain flexible electrode material for electrochemical capacitors. Tian et al. [44] prepared cotton fabric coated with graphene oxide and chitosan via the electrostatic layer-by-layer technique which has ultraviolet (UV) protection property. Tang et al. [9] prepared multifunctional cotton fabric with electrical conductivity and UV protection properties by coating graphene oxide and then synthesizing polyaniline (PANi) by in situ chemical polymerization process. Mizerska et al. [45] obtained the electrical conductivity and hydrophobicity in cotton fabric through a sol-gel method by coating with an organosilicon sol containing graphene oxide followed by reduction of graphene oxide by thermal treatment. Du et al. [46] fabricated graphene based wearable textile strain sensors by coating nonwoven fabric with graphene oxide followed by chemical reduction. Zhou et al. [47] coated cotton fabric with silica (SiO₂)/reduced graphene oxide (RGO) to obtain functional properties such as asymmetric wettability, air permeability and thermal insulating properties.

There is limited information available in the literature about coating spunlace nonwoven fabrics with graphene oxide and imparting multifunctional properties to the nonwoven fabrics. As mentioned above, cotton spunlace nonwoven fabric is environmental friendly, light weight and low cost material. Also, the nonwoven fabric has large surface area and hydrophilic functional groups which makes it an ideal substrate material to use with graphene oxide. The oxygen containing functional groups both from the GO and cotton nonwoven fabric form hydrogen bonds with each other which enhances the adhesion and allows efficient coating. These functional groups make the dip coating process realizable.

Therefore, the aim of this study is the fabrication of cotton spunlace nonwoven fabric by coating with graphene oxide and then by the reduction of graphene oxide with chemical method to obtain multifunctional nonwoven fabric with electrical conductivity. In textile dyeing process, especially in the coloration of cellulose fibers, vat (including indigo) and sulphur dyes are widely used. Currently, sodium dithionite is the most important reducing agent used in the industrial reduction of vat dyes and sulphur dyes [48–50]. Recently, new fields of application have been developed such as chemical reduction of graphene oxide [40, 51–55]. Chemical reduction method is more suitable for GO coated textile materials than the thermal reduction method due to the process temperature lower than 100 °C. Therefore, chemical reduction of graphene oxide coated cotton spunlace nonwoven fabric was carried out with sodium dithionite as reducing agent. To examine the effect of basis weight of nonwoven fabric, cotton spunlace nonwoven fabrics with four different basis weights were coated with GO and reduction reactions applied. Subsequently, the surface morphology and elemental analysis, chemical structure, tensile strength, color coordinates, surface electrical resistance, water contact angle, UV blocking properties were investigated.

2. Experimental

2.1. Materials

The 100% cotton spunlace nonwoven fabric with 4 different basis weights (40, 50, 60, and 70 g/m²) was kindly supplied by Ihsan Sons Limited, Pakistan. The tensile property of the cotton spunlace nonwoven fabrics was indicated in Table 1 according to the data shared by the supplier. All chemicals were of analytical reagent grade and used without further purification. Graphite flakes were purchased from Sigma Aldrich. Hydrogen peroxide (H₂O₂, 35%), sulfuric acid (H₂SO₄, 95%–98%), phosphoric acid (H₃PO₄), potassium permanganate (KMnO₄) and hydrochloric acid (HCl, 37%), sodium dithionite were purchased from Merck. Distilled water was used throughout the experiments.

Table 1. Specifications of cotton nonwoven fabrics with corresponding sample codes.

Sample code	Basis weight (g/m ²)	Tensile strength (MD) (N)	Elongation (%)
NF40	40	23.21 ± 5	28.32 ± 10
NF50	50	35.54 ± 5	33.10 ± 10
NF60	60	38.94 ± 5	34.45 ± 10
NF70	70	48.28 ± 5	34.54 ± 10

2.2. Synthesis of GO

Graphene oxide was synthesized from flake graphite by the improved Hummer's method according to Ref. [56]. Briefly, a 9:1 mixture of concentrated $\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$ (360:40 mL) was added to a mixture of graphite flakes (3 g) and KMnO_4 (18 g). The reaction was then heated to $50\text{ }^\circ\text{C}$ and stirred for 12 h. The reaction was then cooled to room temperature and poured onto ice (400 mL) with 35% H_2O_2 (6 mL). The resulting suspension was washed by repeated centrifugation (each at 8000 rpm for 30 min), first with 400 mL of 1 M HCl and 200 mL of ethanol (2'), then with distilled water until a pH of 4–5 was achieved. The obtained solid product was dried overnight in an oven at $60\text{ }^\circ\text{C}$.

2.3. Coating of cotton nonwoven fabric with GO

The synthesized GO nanosheets were dispersed in distilled water by bath sonication method for about 60 min to obtain 2 mg/mL GO aqueous dispersion. The cotton spunlace nonwoven fabric was dip coated in a bath with liquor to goods ratio (L:G) = 40:1 containing 2 mg/mL GO dispersion for about 30 min at $60\text{ }^\circ\text{C}$. Because of the strong adsorption, the cotton nonwoven was quickly coated by the GO. Then, the coated fabric was kept in an oven at $70\text{ }^\circ\text{C}$ for 30 min. The coating process was repeated 5 times in order to increase the GO adsorption on the nonwoven fabric. The obtained fabrics were coded as GONF according to the basis weights. For example, GONF40 corresponds to the 40 g/m^2 basis weight nonwoven fabric coated with GO.

2.4. Reduction of GO coated cotton nonwoven fabric

The reduction process of graphene oxide coated cotton nonwoven fabrics was carried out using sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) as a reducing agent. The GONF samples were immersed into the reduction bath containing 0.1 M aqueous solution of $\text{Na}_2\text{S}_2\text{O}_4$ for about 60 min. The liquor to goods ratio (L:G) was adjusted as 70:1. The temperature of the reduction process was kept at $95\text{ }^\circ\text{C}$. The resulting fabrics were rinsed with distilled water to remove the remaining reducing agent. The fabrics were dried in an oven at $90\text{ }^\circ\text{C}$. The obtained fabrics after reduction process were coded as RGONF. For example, RGONF40 corresponds to the 40 g/m^2 basis weight nonwoven fabric coated with reduced GO. The GO coating and reduction process is given schematically in Figure 1.

2.5. Characterization

Scanning electron microscopy (SEM, FEI Sirion) was employed to observe the morphology of coated samples. The surfaces of the samples were coated with gold at 1.5 kV for 100 s before analysis. An attenuated total reflection Fourier transform infrared spectrometer (ATR-FTIR, Perkin Elmer Spectrum Two) was used at $450\text{--}4000\text{ cm}^{-1}$. The Raman spectra were acquired using a WITec alpha300 RA with a 523 nm laser (WITech, Germany). The mechanical properties were tested with a tensile mechanical tester (Instron 4411) at the machine direction (MD). It is noteworthy to mention that the almost all nonwoven fabrics are anisotropic, having more fiber orientation in the machine direction (MD) than the cross direction (CD) [57]. The specimens were prepared at dimensions of $75 \times 50\text{ mm}$. The crosshead speed was 300 mm/min. Each sample was tested at least five times and the average value was calculated. The tensile strength and elongation at break were determined. Color coordinates were determined by Datacolor SF600+, using SAV aperture and SI mode, and the color differences were calculated in accordance with the CIELab system with D65/10° observer values. In CIE Lab system, the lightness L^* represents the darkest black at $L^* = 0$ and the brightest white at $L^* = 100$, a^* represents redness (a^* is +)/greenness (a^* is -) and b^* represents yellowness (b^* is +)/blueness (b^* is -). a^* and b^* represent true neutral gray values at $a^* = 0$ and $b^* = 0$. The color difference (ΔE) was determined using the following equation [9]:

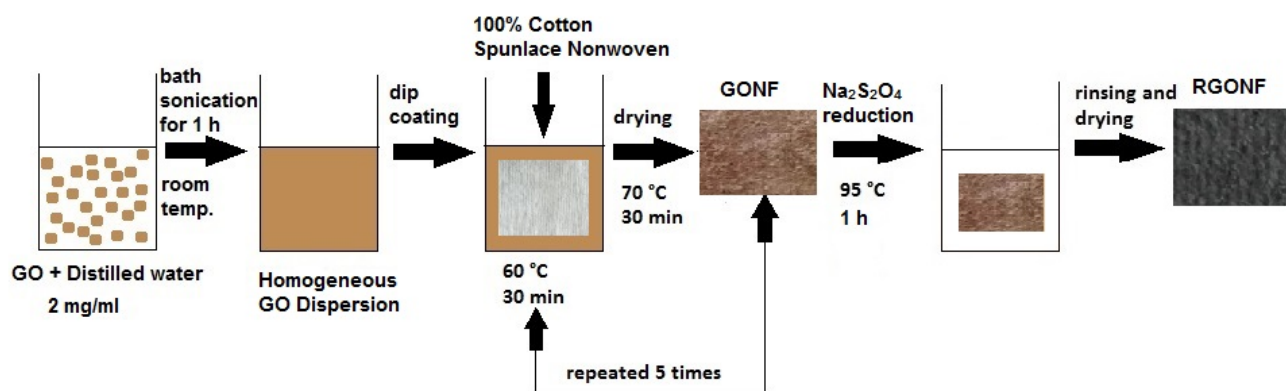


Figure 1. Coating and reduction processes of GO onto cotton nonwoven fabric.

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}. \quad (1)$$

The surface electrical resistance of the fabrics was measured using a standard four-point probe.

The setup consists of a sourcemeter (Keithley 2450 Sourcemeter) and a four point probe station (Everbeing Int'l Corp). Each sample was measured at least five times and the average value was calculated. The water contact angle of the cotton nonwoven fabrics was measured by using of a PGX Goniometer (FIBRO Systems, Sweden). In the measurements, deionized water was used as standard liquid that was deposited on the nonwoven fabric surface. The UV protection ability was recorded by a UV transmittance analyzer (UV 1000F, Labsphere, SDL ATLAS). Each sample was measured at least five times and the average value was calculated. The protection activity of a fabric to block ultraviolet light is defined as the ultraviolet protection factor (UPF) value. The test was done under the wavelength range of 290–400 nm. The mean UPF value and UVA and UVB transmittance of GONF and RGONF coated fabrics were calculated according to the EN 13758-1:2001 [58, 59]. UPF was calculated as follows:

$$UPF = \frac{\int_{290}^{400} E_{\lambda} \times \epsilon_{\lambda} \times d\lambda}{\int_{290}^{400} E_{\lambda} \times \epsilon_{\lambda} \times T_{\lambda} \times d\lambda} \quad (2)$$

where E_{λ} is the solar irradiance, ϵ_{λ} is the erythema action spectrum, T_{λ} is the spectral transmittance of the specimen (incoming light that passes through the specimen), $d\lambda$ is the wavelength increment (nm), and λ is the wavelength (nm).

The UPF rating was calculated as follows:

$$UPF = UPF_{\text{average}} - t_{\alpha/2, n-1} \frac{s}{\sqrt{n}}, \quad (3)$$

where s is the standard deviation, n is the number of the specimens and $t_{\alpha/2, n-1}$ is the table value for $\alpha = 0.05$. A fabric having UPF rating higher than 40 (UPF 40+) can be labelled as UV protective. The durability of electrical surface resistivity, water contact angle and UV protection against to the washing was performed according to ISO 105 C06-A1S.

3. Results

3.1. Surface morphology and elemental analysis

The SEM images of neat cotton nonwoven, GO coated cotton nonwoven and RGO coated cotton nonwoven fabric were presented in Figure 2. From Figure 2a, it can be seen that the surface of the neat cotton fibers are smooth and the typical longitudinal fibril structure with some convolutions along its length is evident. After coating with GO, the cotton fibers are covered by the GO sheets which appears as a wrinkled and rough surface on the fibers (Figure 2b). Some RGO sheets could be seen on cotton fibers after reduction process (Figure 2c). RGO sheets covered the cotton fibers and stacked to each other. The wrinkled and rough structure of RGO sheets on cotton fibers were remained, indicating the presence of reduced nature of GO sheets on fibers [60]. Moreover, the roughened surface composed of micropores is important for the hydrophobicity [61]. Furthermore, the elemental analysis of the neat cotton nonwoven fabric and RGO coated cotton nonwoven fabric were shown in Figures 2d and 2e, respectively. Also, the atomic and weight percentage ratios of neat cotton and RGO coated cotton nonwoven fabrics were presented in Table 2. The results show that the neat cotton nonwoven fabric composed of C and O having the atomic percentage ratio of 62.01% and 37.99%, respectively. However, the atomic percentage ratio of C and O for the cotton nonwoven fabric changed after the reduction process of GO. The atomic percentage ratio of C and O for the RGO coated nonwoven fabric was found as 68.44% and 31.56%, respectively. This results indicates that the C/O ratio increased with the reduction of GO, showing the formation of RGO sheets on cotton fibers.

3.2. FTIR spectroscopy

The FTIR spectra of the neat cotton (NF70), GO coated (GONF70) and RGO coated (RGONF70) cotton nonwoven fabrics were given in Figure 3. The FTIR spectra of the neat cotton nonwoven fabric shows all the characteristic peaks of pure cotton, including hydrogen bonded OH stretching at 3333–3270 cm^{-1} , asymmetrical C-H stretching at 2894 cm^{-1} , O-H bending at 1638 cm^{-1} , CH_2 symmetric bending at 1426 cm^{-1} and the C-H bending at 1359 cm^{-1} [16, 62]. However, some changes in the FTIR spectra occurred after the GO coating. A new peak appears at the 1743 cm^{-1} which corresponds to C=O stretching arising from the carboxyl groups and the carbonyl containing groups in the GO [14, 62, 63]. The C-H stretching peak at 2894 cm^{-1} shifted to 2921 cm^{-1} and a new peak appeared at 2850 cm^{-1} because of the p-p conjugation between cotton and GO which leads to decreased bond strength. This indicates that the cellulose macromolecules chains were subjected to minor changes in intra and/or inter hydrogen bonding during GO coating and reduction processes [64, 65]. The peaks changed after the reduction process. The intensity of peak at 1638 cm^{-1} was decreased in the RGO coated

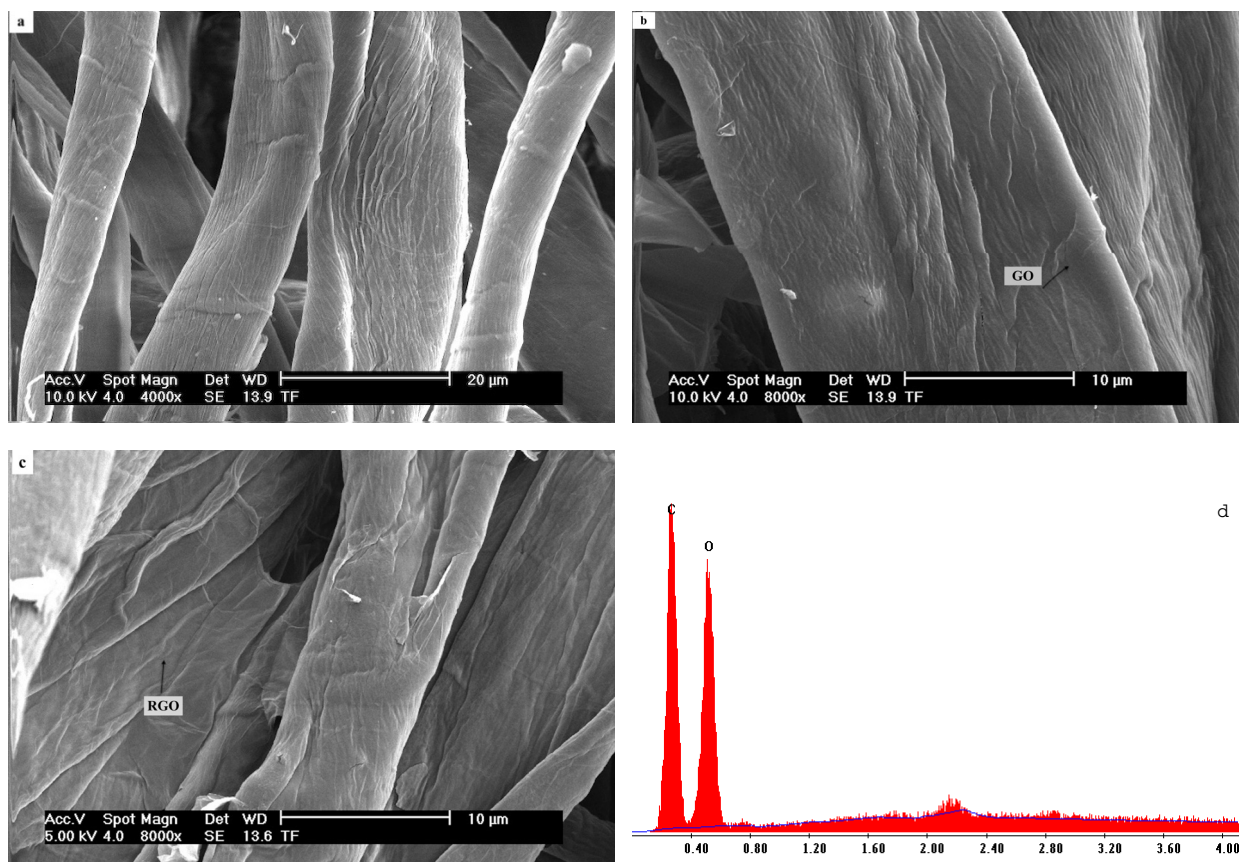


Figure 2. SEM images of cotton nonwoven fabric (a), GONF70 (b), RGONF70 (c), EDS spectra of cotton nonwoven fabric (d) and RGONF70 (e).

Table 2. Elemental analysis of cotton nonwoven and RGO coated nonwoven fabric obtained by EDS.

Sample	Atomic ratio (%)		Weight ratio (%)	
	Elements		Elements	
	C	O	C	O
NF	62.01	37.99	55.06	44.94
RGONF	68.44	31.56	61.95	38.05

cotton nonwoven fabric as compared to the neat cotton nonwoven fabric. Also, the peak at 1743 cm^{-1} became narrower than that of the GO coated cotton nonwoven fabric indicating the GO was successfully reduced into the RGO after the chemical reduction process with $\text{Na}_2\text{S}_2\text{O}_4$ [16]. A broad peak appeared at 1553 cm^{-1} is attributed to C=C skeletal vibration of graphene [43].

3.3. Raman analysis

The structural changes before and after GO coating and after reduction process of GO were further characterized by Raman analysis. Raman spectra of cotton spunlace nonwoven fabric, GO and RGO coated sample are given in Figure 4. The spectrum of cotton spunlace nonwoven fabric (NF70) shows the characteristic peaks assigned to cotton. The strong peak at 1099 cm^{-1} is assigned to the asymmetric vibration of glycoside links in the cotton nonwoven fabric [66]. After GO coating process, the spectrum of cotton nonwoven changed prominently and the presence of GO nanosheets onto the cotton fibers can be easily differentiated by two characteristic peaks of carbon materials. The GONF70 and RGONF70

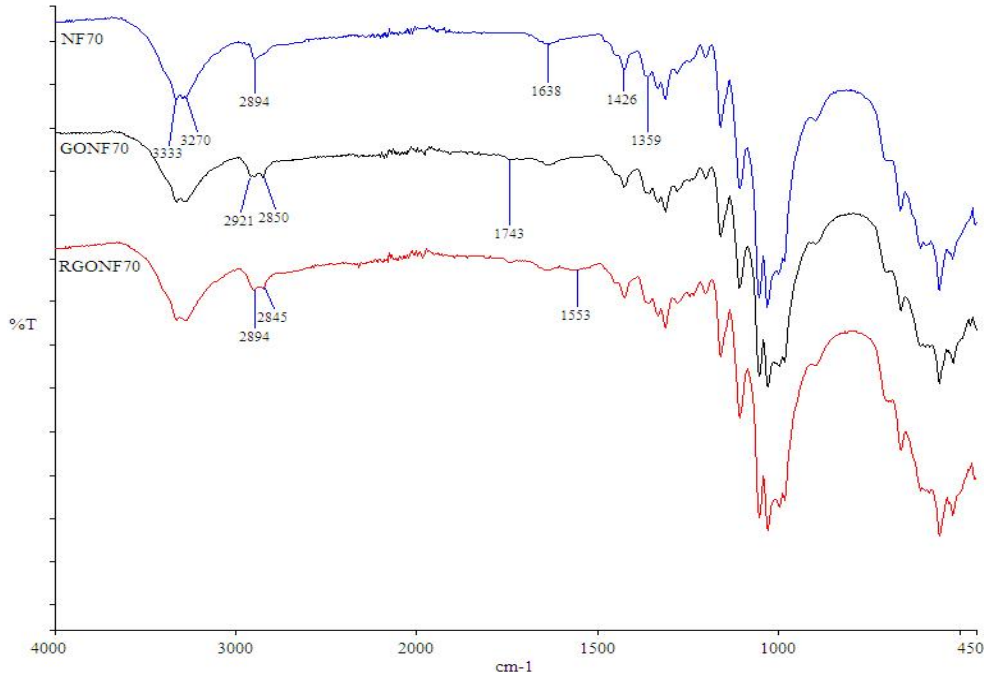


Figure 3. FTIR spectra of NF70, GONF70 and RGONF70.

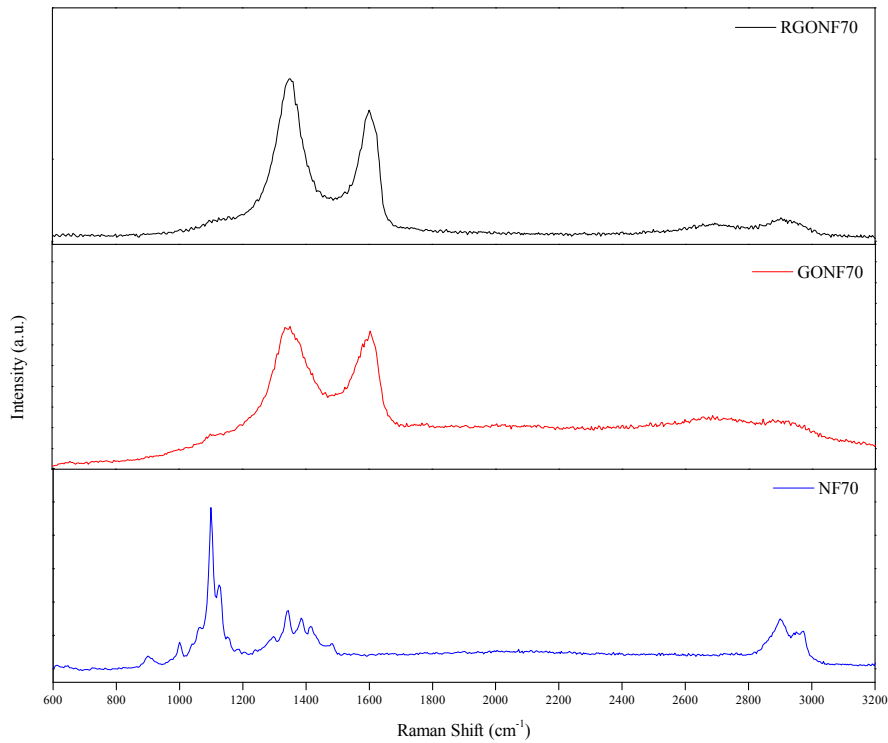


Figure 4. Raman spectra of NF70, GONF70 and RGONF70.

samples exhibit two prominent peaks correspond to the D band and G band, indicating the main characteristics of carbon materials, which reveal the coating of GO and RGO onto nonwoven fabric. From the spectra, it can be seen that the D band is located at 1351 cm^{-1} for both GONF70 and RGONF70 samples. G band is located at 1604 cm^{-1} for GONF70 and at 1600 cm^{-1} for RGONF70. The intensity ratio of D and G band (I_D/I_G) for GONF70 and RGONF70 was obtained as 1.02 and

1.07, respectively. The intensity of D band is higher than the G band of RGONF70 sample which reveals the presence of defects. It can be understood that not only the oxidation but also the chemical reduction process using sodium dithionite contributed to an increase of the structural disorder. The increase in I_D/I_G ratio after reduction suggests a decrease in the average size of the sp^2 domains upon the removal of oxygen containing functional groups [14, 67, 68].

3.4. Mechanical property

The mechanical performance of GO coated and RGO coated cotton nonwoven fabrics were tested in terms of tensile strength and elongation properties in the machine direction (MD). The detailed results are listed in Table 3. The tensile strength and elongation of the neat cotton nonwoven, GO coated cotton nonwoven and RGO coated cotton nonwoven fabrics is given in Figures 5 and 6, respectively. In Figure 5, it can be seen that the tensile strength of GO coated cotton nonwoven fabric decreased in comparison with the neat cotton nonwoven fabric. After the reduction process is applied to GO coated cotton nonwoven fabric, the decrease in tensile strength still continued. The highest tensile strength value was obtained at the GONF70 as 44.89 N showing percent change as low as 7.02%. The highest tensile strength value after reduction was obtained at the RGONF60 as 9.43 N. It can be seen from the tensile strength values of reduced samples, the

Table 3. Tensile strength and elongation of GO and RGO coated cotton nonwoven fabrics.

Sample code	Tensile strength (MD) (N)	Tensile strength (MD) (cN/tex)	Elongation (%)
After coating			
GONF40	14.30 ± 0.760	0.64 ± 0.024	27.89 ± 2.667
GONF50	26.11 ± 1.543	0.69 ± 0.010	31.84 ± 2.613
GONF60	22.86 ± 0.201	0.65 ± 0.026	31.84 ± 3.953
GONF70	44.89 ± 1.077	1.03 ± 0.026	27.88 ± 1.769
After reduction			
RGONF40	4.13 ± 0.725	0.21 ± 0.027	28.75 ± 4.428
RGONF50	8.03 ± 0.696	0.28 ± 0.023	31.65 ± 6.582
RGONF60	9.43 ± 0.532	0.32 ± 0.004	33.85 ± 3.767
RGONF70	3.82 ± 0.574	0.11 ± 0.020	27.58 ± 2.303

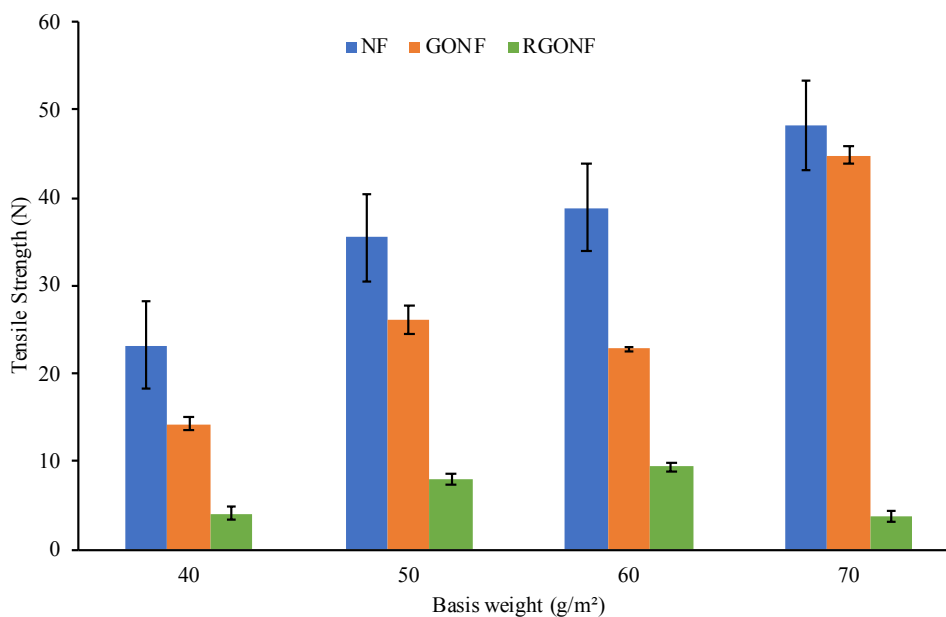


Figure 5. Tensile strength of cotton nonwoven, GO and RGO coated cotton nonwoven fabrics.

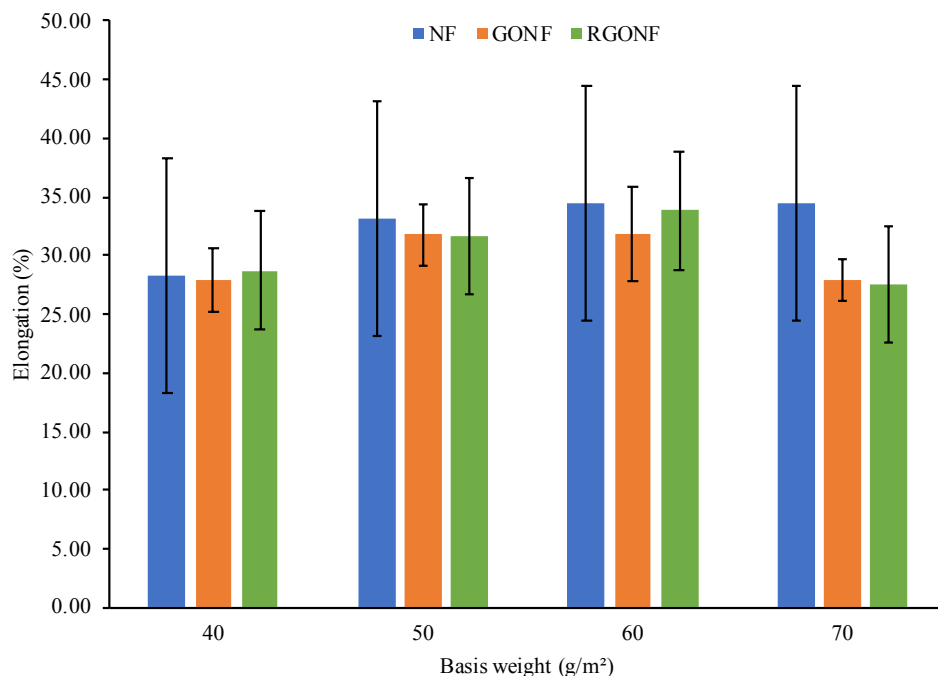


Figure 6. Elongation of cotton nonwoven, GO and RGO coated cotton nonwoven fabrics.

mechanical performance of spunlace cotton nonwoven fabric coated with graphene oxide nanosheets was become weaker with the further reduction treatments. The explanation for the obtained results can be attributed to the poor bonding characteristics of the spunlace nonwoven structure. The basic building material that constitute the nonwoven fabric used in this study is the short staple cotton fibers and these fibers have become nonwoven fabric by mechanically bonding with the help of water jets. Since the structural integrity of the fabric is disrupted after each wet treatment, heating and mechanical effect applied, there has been a significant decrease in the mechanical properties.

On the other hand, the elongation of cotton spunlace nonwoven fabrics after GO coating showed little decrease at GONF40, GONF50 and GONF60 with percent change of 1.51%, 3.80% and 7.57%. The highest decrease in elongation after GO coating process is obtained at GONF70 with percent change of 19.28%. Besides, with the reduction of GO nanosheets coated on the cotton spunlace nonwoven fabrics, better elongation results were obtained. The elongation of RGONF40 and RGONF60 increased from 27.89% and 31.84% to 28.75% and 33.85%, respectively.

In order to determine the effect of GO coating and reduction process on the mechanical properties of cotton spunlace nonwoven fabric, statistical analysis was performed. One way ANOVA test was conducted for the mechanical properties of the nonwoven fabric samples at significance level of 0.05. Table 4 indicates the variance analysis results of GONF and RGONF samples. According to the results, GO coating and reduction process had significant effect on tensile strength of cotton spunlace nonwoven fabric. However, it was found out that the effect of GO coating of NF70 sample on the tensile strength was not statistically important. On the other hand, GO coating and reduction process were found out as no significant effect on elongation of cotton spunlace nonwoven fabric.

3.5. Color coordinates and reflectance measurements

As an easy way for determination of deposition and reduction of GO, the color of the fabric can be considered. The color changes of the cotton nonwoven fabrics after GO coating and reduction processes were determined by reflectance spectra and color coordinates. The reflectance spectra of cotton nonwoven fabric, GO and RGO coated cotton nonwoven fabrics within 360–700 nm are given in Figure 7. The reflectance spectra of the neat cotton nonwoven fabrics show that the fabrics reflect the most of the light in the visible region. The common characteristics for the neat cotton nonwoven fabrics are the high reflectance values across all wavelength which imply the materials are white. For the cotton nonwoven fabrics coated with GO, the reflectance curves of the fabrics shift to lower values which indicates that the most of the light is absorbed by the fabric and the color becomes darker. However, the spectra of GO coated nonwoven fabrics shows a tendency to increase with high wavelengths between 550 and 700 nm. This indicates that the GO coated cotton nonwoven fabrics are reflecting the light in yellow region (560–590 nm), orange region (590–620 nm) and red region (620–700 nm). In the case

Table 4. One way ANOVA results for tensile strength and elongation of cotton nonwoven fabrics.

Source		Tensile strength (N)	Elongation (%)
GONF	40	0,04*	0,95
	50	0,03*	0,84
	60	0,00*	0,69
	70	0,28	0,32
RGONF	40	0,00*	0,95
	50	0,00*	0,84
	60	0,00*	0,93
	70	0,00*	0,31

*statistically important according to $\alpha = 0.05$.

of RGO coated cotton nonwoven fabrics, the reflectance spectra show curves which are essentially flat at approximately 5% denoting the absorption of the light in all wavelengths. This reveals that RGO coated cotton spunlace fabrics absorb the most of the incident light without showing a distinct peak at the visible region. It can be understood that RGO coated cotton spunlace fabrics had dark color close to black.

The color coordinates and the color differences of the cotton nonwoven fabrics were given in Table 5. From the results, it can be seen that the cotton nonwoven fabrics have the highest L^* values showing the bright whiteness, which increases with the increase in basis weight. After the coating process of cotton nonwoven fabrics with GO, the L^* values decrease which indicates that the color of the nonwoven fabric becomes darker. The coating with GO changes the color of the nonwoven fabric from white to yellow-brown and this indicates the deposition of GO sheets on the cotton nonwoven fabric. According to the data, the L^* value of the cotton nonwoven fabric with 70 g/m² basis weight decreased from 94.35 to 51.41 after GO coating. There were also changes in a^* , b^* and C^* values of fabrics. The coating of the cotton nonwoven fabrics with GO causes an increase in a^* values towards redness with the increase in basis weight. Also, the significant increase in b^* value indicates the increase in the yellowness. Furthermore, the b^* value increases with the increase in basis weight from 40 to 70 g/m² and the highest yellowness value is obtained with the highest basis weight which is GONF70. Also, the C^* value increases for all GO coated nonwoven fabrics indicating that the color turned to yellow-brown. However, when the reduction process takes place, the a^* , b^* and C^* values of all of the fabrics decrease dramatically. The L^* value of the RGONF70 sample decreased to 31.07 indicating the blackness in the color. The a^* , b^* and C^* values of the RGO coated cotton nonwoven fabrics decrease to almost zero which indicates that there are no dominant color and the fabrics are neutral grey. Additionally, the ΔE values show the color differences of the GO coated and reduced GO coated cotton nonwoven fabrics compared to neat cotton nonwoven fabric. The increased ΔE value of the GO coated nonwoven fabrics shows high degree of color difference from the neat cotton nonwoven fabric. However, after the reduction process the ΔE value of the RGONF70 sample increases to 63.284 which denotes the increase in the color difference compared to the neat cotton nonwoven fabric. In conclusion, the obtained data from the reflectance spectra and the color coordinates simultaneously show that the GO sheets are successfully and effectively deposited on the cotton nonwoven fabric and reduced.

3.6. Electrical surface resistance

The electrical surface resistance values of RGO coated cotton nonwoven fabrics before and after washing were given in Figure 8. The electrical conductivity is the prominent feature of the extent to which GO is transformed to RGO by the reduction process. The neat cotton and GO coated nonwoven fabrics have an insulating characteristic. After the reduction of GO coated nonwoven fabric by sodium dithionite, the surface resistance of fabrics as a function of basis weight was measured. The electrical surface resistance of RGO coated cotton nonwoven fabric before washing decreased from $1.16 \times 10^3 \Omega/\text{sq}$ to $5.98 \times 10^2 \Omega/\text{sq}$ by the increase of basis weight of the nonwoven fabrics from 40 g/m² to 70 g/m². The basis weight of the nonwoven fabric plays an important role for the enhancement of the electrical conductivity of RGO coated cotton nonwoven fabrics. The decrease in surface resistance could be explained by the increase of the nonwoven fabric weight. Nonwovens have porous structure which affects their mechanical, thermal and comfort properties. With the increase in basis weight and thickness of the nonwoven fabric, the more number of fibers participate in entanglement, thus,

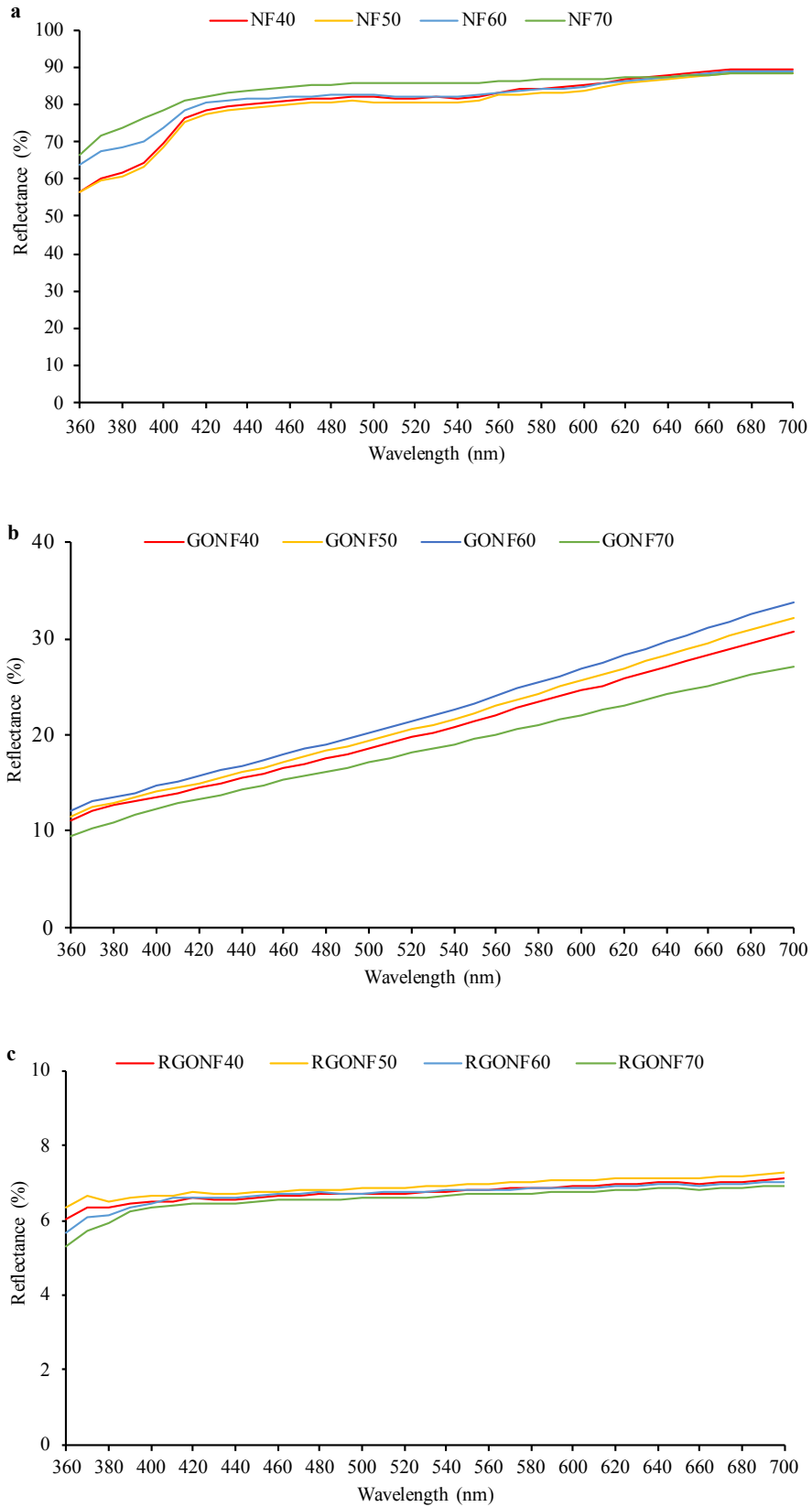
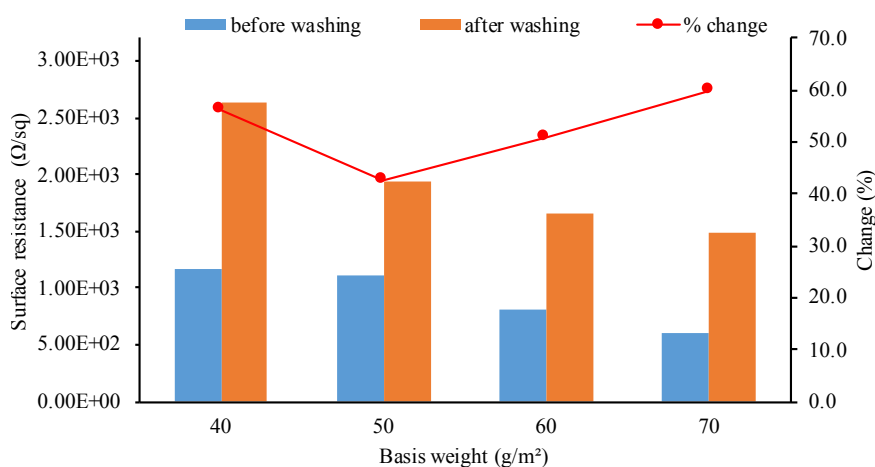


Figure 7. Reflectance spectra of the cotton nonwoven fabric (a), GO coated cotton nonwoven fabric (b) and RGO coated cotton nonwoven fabric (c).

Table 5. Color coordinates and color differences of cotton nonwoven, GO coated cotton nonwoven and RGO coated cotton nonwoven fabrics.

Sample	L*	a*	b*	C*	h	X	Y	Z	ΔE
NF40	93.07	1.48	2.03	2.51	53.97	79.56	83.13	86.36	-
NF50	92.57	1.50	2.00	2.50	53.10	78.48	81.99	85.20	-
NF60	93.17	1.33	1.33	1.88	45.18	79.71	83.37	87.58	-
NF70	94.35	0.11	1.44	1.45	85.53	81.67	86.08	90.29	-
GONF40	53.62	3.76	11.17	11.78	71.40	21.28	21.62	17.31	40.558
GONF50	54.57	3.88	11.39	12.04	71.20	22.17	22.51	17.99	39.220
GONF60	55.61	4.04	11.58	12.27	70.79	23.20	23.53	18.79	39.028
GONF70	51.41	3.17	10.26	10.74	72.83	19.22	19.62	15.96	43.940
RGONF40	31.36	0.29	0.76	0.81	68.90	6.48	6.80	7.10	61.742
RGONF50	31.71	0.28	0.79	0.84	70.56	6.62	6.96	7.25	60.886
RGONF60	31.36	0.19	0.56	0.59	71.34	6.47	6.81	7.16	61.827
RGONF70	31.07	0.21	0.72	0.75	73.48	6.35	6.68	6.98	63.284

**Figure 8.** Electrical resistivity of RGO coated cotton nonwoven fabrics before and after washing.

the interfiber space is closed. Also, basis weight affects the porosity of nonwoven fabric. The increase in basis weight causes the decrease in pore size of the spunlace nonwoven fabric [26]. Hence, the RGO sheets covers the surface of constituent fibers and inter-fiber spaces uniformly. Consequently, the decrease in surface resistance could be attributed to the effective recovery of the sp^2 network of carbon via chemical reduction and good RGO sheet-to-sheet connection throughout the nonwoven fabric. In other words, RGO nanosheets form a continuous conductive thin layer on the surface of the spunlace nonwoven fabric to shorten the electron transfer pathways [64, 69].

The electrical surface resistance of RGO coated nonwoven fabrics after washing increased slightly. The surface resistance of nonwoven fabrics after washing shows a similar trend like before washing. The increase in surface resistance after washing can be explained by the removal of loosely adsorbed RGO sheets. The change in percentage in surface resistance values of RGO coated cotton nonwoven fabrics before and after washing was also calculated. According to the results, the lowest % change was obtained with the nonwoven fabric having basis weight of 50 g/m². This can be explained by the better coating of the GO sheets on the surface of cotton fibers, thus, less amount of RGO sheets removed during the washing process. However, the disruption of structural integrity of cotton spunlace fabric with wet processes has also affected the percentage change in surface electrical resistance. Due to the low dimensional stability and further loosening

of the entanglement of staple cotton fibers after washing process, the interfiber space has widened, thus, the electrical pathway of RGO sheets was damaged. The reason of the higher % change in RGONF70 sample than the others could be the more deformation of dimensional stability and disruptions of fibers during wet processes. Besides, the lowest electrical surface resistance after washing was obtained as $1.49 \times 10^3 \Omega/\text{sq}$ for RGONF70 sample. Furthermore, Figure 9 shows the electrical conductivity of RGO coated cotton nonwoven fabric by integrating with the red LED light. The RGO coated cotton nonwoven fabric has potential as an electrical conductor in various functional application areas.

3.7. Water contact angle

Figure 10 shows the water contact angle of GO and RGO coated cotton nonwoven fabrics with basis weight of 70 g/m^2 . Figure 11 shows the digital photographs of the water droplets placed on the surface of the cotton nonwoven fabric, GO and RGO coated cotton nonwoven fabrics with basis weight of 70 g/m^2 . Cotton is hydrophilic in nature and has good water absorption property [16]. The neat cotton nonwoven fabric can be completely wetted by water due to the abundant hydroxyl groups in its structure (Figure 11) [7]. Also, the GO coated cotton nonwoven fabric shows a similar behavior due to the presence of sufficient oxygenated functional groups on the basal planes and edges of the GO sheets [70]. The GO coated cotton nonwoven fabric (Figure 10) shows hydrophilicity with an average water contact angle of

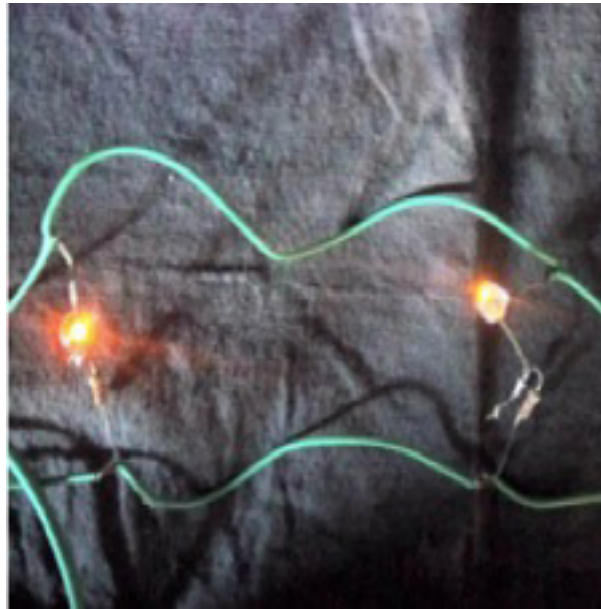


Figure 9. Digital photograph of a red LED light integrated with RGO coated cotton nonwoven fabric.

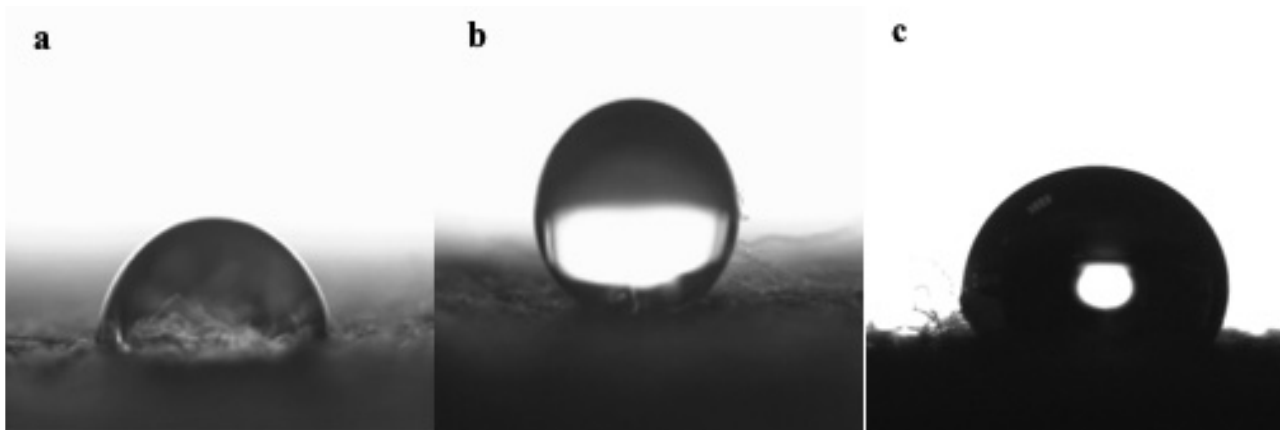


Figure 10. Water droplet images of GONF70 (a), RGONF70 before (b) and after washing (c).

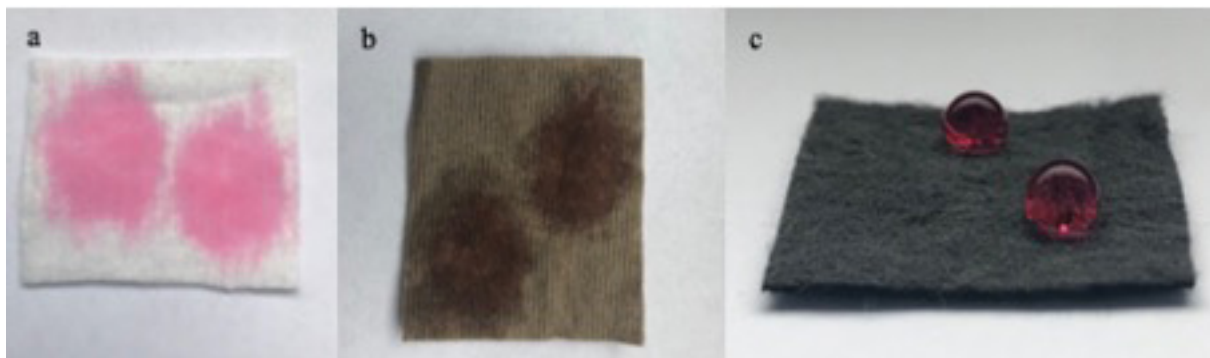


Figure 11. Digital image of colored water droplets on the cotton nonwoven fabric (NF70) (a), GO coated nonwoven fabric (GONF70) (b), RGO coated cotton nonwoven fabric (RGONF70) (c).

56.1°. When the reduction process performed, the change in water contact angle is remarkable. The water contact angle of RGO coated cotton nonwoven fabric increases to 108.2° with resulting hydrophobicity. The increase in water contact angle can be explained by the elimination of oxygen-containing functional groups after reduction. The phenomenon of the hydrophobicity can be referred as the contact angle value bigger than 90° [71]. After washing process, the water contact angle of RGONF70-W sample obtained as 106.6°. It is found out that the hydrophobic behavior of RGONF70 sample sustained after washing.

The water droplets placed on the surface of the RGO coated cotton nonwoven fabric are stable and could maintain their spherical shapes for a long period of time (Figure 11). Hydrophobic behavior of the RGO coated cotton nonwoven fabric can be explained by the removal of oxygen containing functional groups during reduction process.

3.8. UV protection property

The ultraviolet transmittance spectra of the cotton nonwoven fabrics, GO and RGO coated cotton nonwoven fabrics between 250 and 450 nm wavelength are given in Figure 12. In Figure 12a, the UV transmittance spectra of cotton spunlace nonwoven fabrics can be seen. The UV transmittance of neat cotton nonwoven fabrics has high values and gives different results depending on the basis weight. The highest UV transmittance was found at NF40 which has the lowest basis weight. With the increase in basis weight, UV transmittance decreases. Nevertheless, the UV transmittance of neat cotton spunlace nonwoven fabrics has high values throughout the UV region. Also, the UV transmittance curves of all fabrics show increasing trend with the increase of wavelength. The decline in UV transmittance can be seen in GO coated nonwoven fabric (Figure 12b). From the spectra, it is demonstrated that the coating of GO sheets acted as an effective material to block UV rays across fabric. UV rays can easily penetrate to the neat cotton nonwoven fabric whereas the penetration of UV rays are blocked considerably by the GO sheets on the cotton nonwoven fabric. Especially, GO coated cotton spunlace nonwoven fabrics with higher basis weights have lower UV transmittance percentage values in comparison with GO coated cotton spunlace nonwoven fabric with basis weight of 40 g/m². The lowest UV transmittance values were obtained with GONF70. The UV transmittance spectra of RGO coated cotton spunlace nonwoven fabrics were given in Figure 12c. It can be seen that the homogenous coating of GO and successfully transformation of GO to reduced GO have affected the UV transmittance of the cotton spunlace nonwoven fabric in a positive way. Moreover, after the reduction process, the RGO coated cotton nonwoven fabric had much lower UV transmittance compared to neat and GO coated cotton nonwoven fabric. The attenuation in RGO coated cotton nonwoven fabric can be attributed to the UV absorption ability of reduced graphene oxide nanosheets. However, the basis weight of cotton spunlace nonwoven fabric has a prominent effect on UV transmittance. It can be obviously said that the higher the basis weight, the lower the UV transmittance. It is noteworthy to mention that the UV transmittance curves of the RGO coated cotton spunlace nonwoven fabrics became straighter without showing an increasing trend throughout the scanned UV region wavelengths.

In order to assess the degree of UV protection of cotton nonwoven, GONF, RGONF and RGONF-W samples as a function of the basis weight, the ultraviolet protection factor (UPF) was measured and given in Figure 13. The neat cotton spunlace nonwoven fabrics had the lowest UPF values. However, the UPF value showed a slight increase with the increase in basis weight of the cotton spunlace nonwoven fabrics. For the cotton nonwoven fabrics coated with GO, with the sequential increase in basis weight as 40, 50, 60 and 70 g/m², the UPF values increased to 9.3, 33.35, 37.59 and 59.2, respectively. Fabric construction and composition are important parameters to the assessment of UV protection. As higher the basis weight of the nonwoven fabric, higher the thickness of the nonwoven fabric. The increase in thickness affects

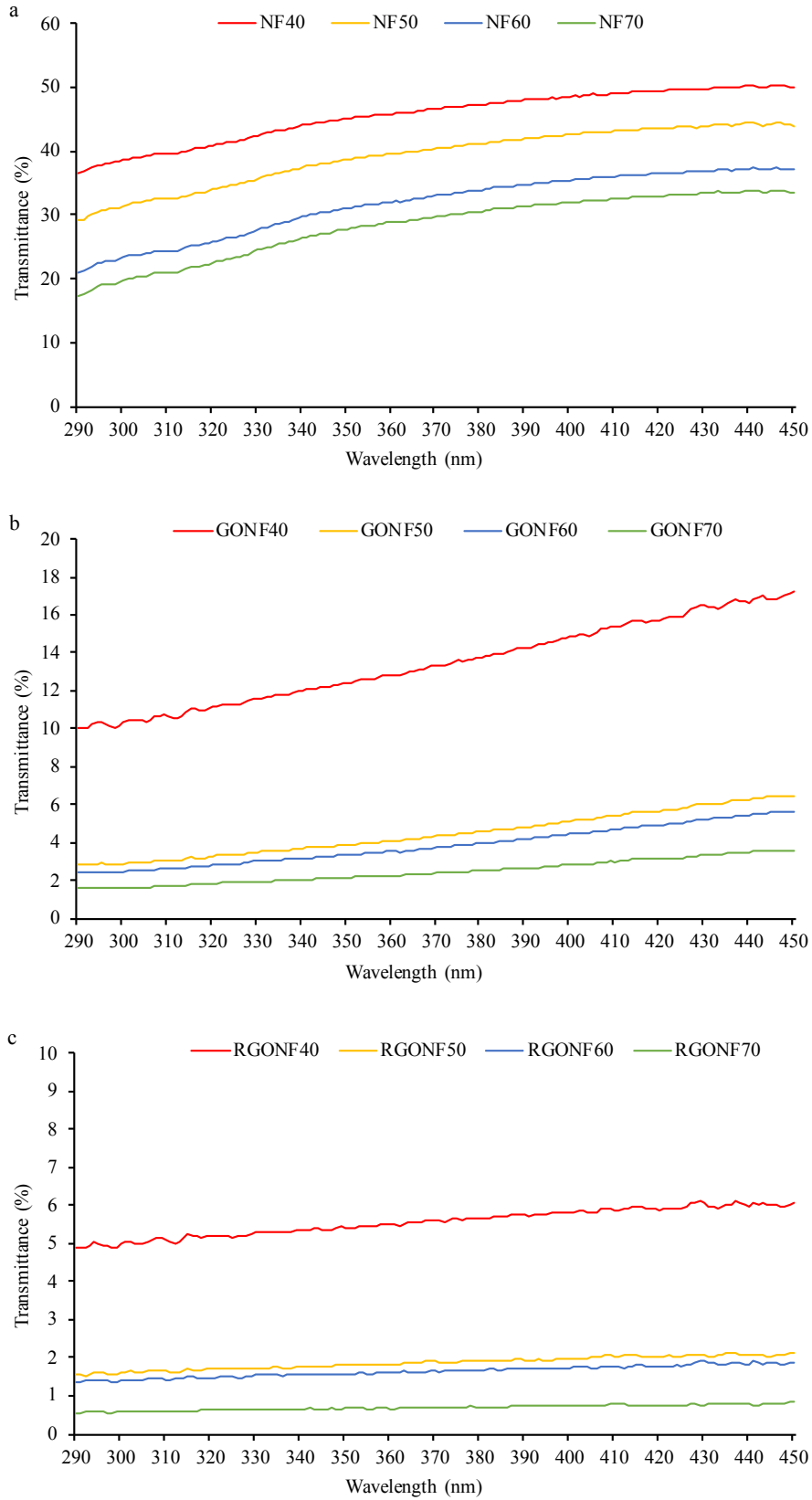


Figure 12. UV transmittance spectra of cotton nonwoven fabrics (a), GO coated cotton nonwoven fabrics (b) and RGO coated nonwoven fabrics (c).

the ability of UV protection in a positive way. When the reduction process is applied to the GO coated cotton nonwoven fabrics, the highest UPF values as a function of basis weight were obtained. The UPF value of the RGONF40 is 19.77 while the UPF value of the RGONF70 is 167.34 which has an excellent protection with a UPF rating 40+ (according to the EN 13758-1:2001). After washing process, UPF values of RGONF samples showed a slight decrease. Also, UPF values of RGONF-W samples change in direct proportion with the increase in basis weight. The UPF values for RGONF40-W, RGONF50-W, RGONF60-W and RGONF70-W samples obtained as 13.81, 55.65, 62.15 and 149.18, respectively. The UPF ratings of cotton nonwoven, GONF, RGONF and RGONF-W samples as a function of basis weight according to the EN 13758-1:2001 standard are listed in Table 6. As it can be seen that RGO coated cotton spunlace nonwoven fabrics at higher basis weight which were coded as RGONF60 and RGONF70 have excellent UV protection property. After washing, the UPF ratings show slight decrease for RGONF40 and RGONF50 samples. On the other hand, the higher basis weight samples continued to show excellent UV protection after washing.

The transmittance of UVA and UVB rays of the cotton nonwoven, GONF, RGONF and RGONF-W samples were shown in Figure 14. The neat cotton nonwoven fabrics have the highest transmittance percentage for both UVA and UVB rays that decrease with the increase in basis weight. For the GO coated cotton nonwoven fabrics, the transmittance percentage for both UVA and UVB decreased prominently compared to the neat cotton nonwoven fabric (Figure 14a). Besides, the sharp decrease in transmittance percentage can be seen in samples GONF40 and GONF50 as a result of increase in basis weight. The decrease in transmittance percentages for both UVA and UVB rays further continued with increase of basis weight of cotton spunlace nonwoven fabric to 70 g/m². On the other side, from Figure 14b it can be seen that the RGO coated cotton spunlace nonwoven fabrics had the lowest transmittance percentage in both UVA and UVB which imply the high level of blocking the penetration of UV rays through the fabrics. The UVA and UVB transmittance percentages of RGO coated cotton spunlace nonwoven fabrics were obtained below 5% for RGONF50, RGONF60 and RGONF70 and

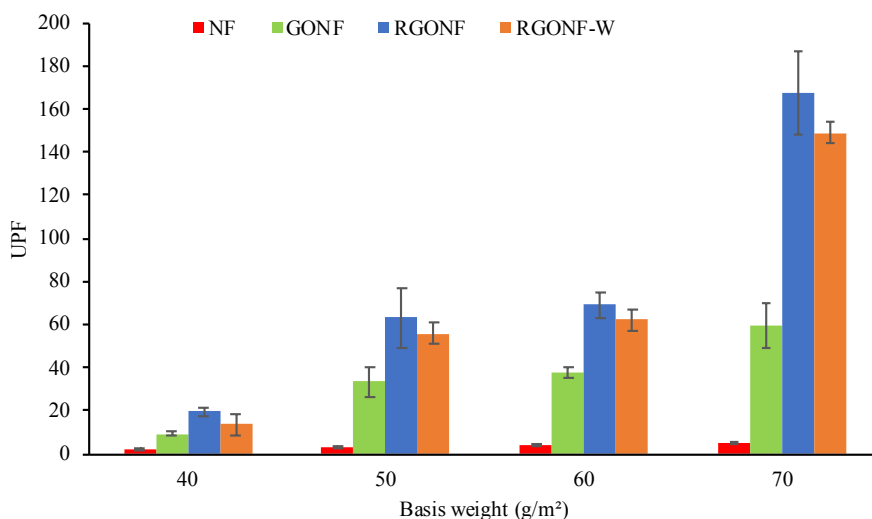


Figure 13. UPF values of cotton nonwoven fabrics, GO coated cotton nonwoven fabrics and RGO coated cotton nonwoven fabrics before and after washing as a function of basis weight.

Table 6. UPF ratings of cotton nonwoven fabrics, GO coated cotton nonwoven fabrics and RGO coated cotton nonwoven fabrics as a function of basis weight.

Sample code	UPF rating (EN 13758-1:2001)			
	NF	GONF	RGONF	RGONF-W
40	2.32	8.61	17.61	12.74
50	2.67	26.56	48.19	47.16
60	3.71	34.67	50+	50+
70	4.27	48.45	50+	50+

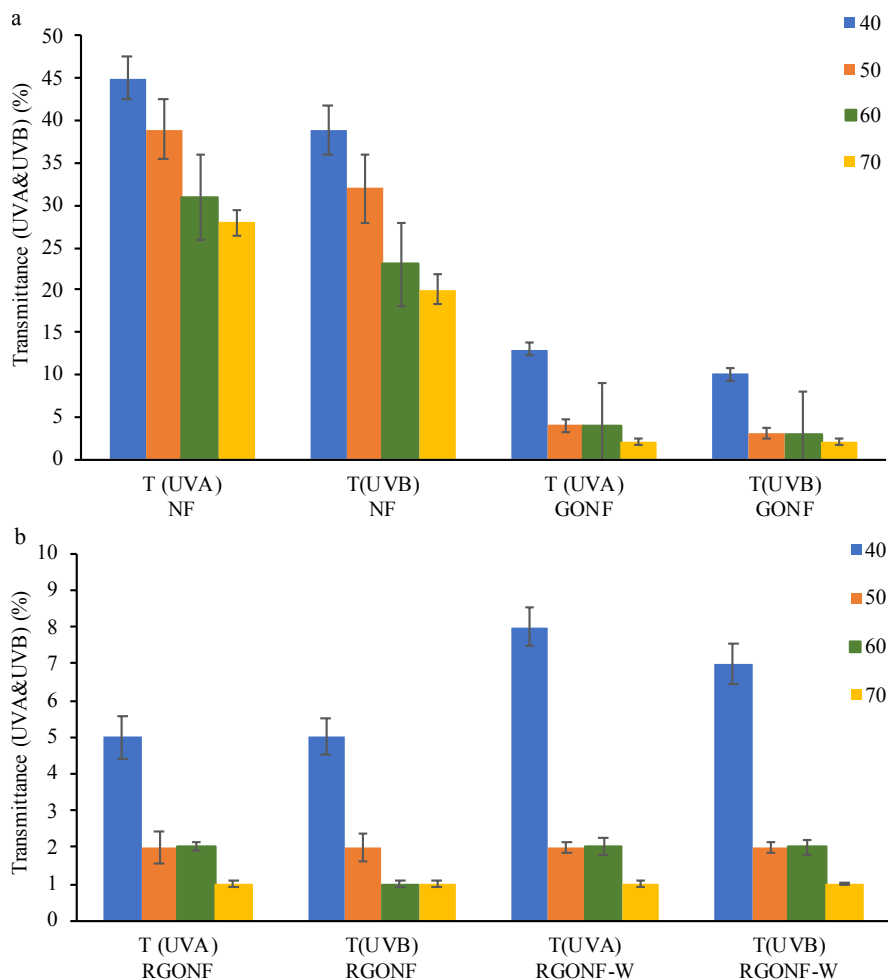


Figure 14. UVA & UVB transmittance of cotton nonwoven fabrics, GO coated cotton nonwoven fabrics (a) and RGO coated cotton nonwoven fabrics before and after washing (b) as a function of basis weight.

at 5% for RGONF40. After washing, the transmittance percentage of RGONF-W samples increased slightly. The lowest transmittance percentage was obtained with the highest basis weight sample in consistent with above mentioned results.

4. Discussion

Cotton spunlace nonwoven fabrics were successfully coated with GO nanosheets and chemical reduction process were applied with aqueous solution of sodium dithionite. The effect of basis weight of the cotton spunlace nonwoven fabrics on the properties of final material was investigated. Mechanical properties, color coordinates and color differences, surface electrical resistance, water contact angle and UV blocking property are studied. It is revealed that the basis weight of the cotton nonwoven fabrics has an improving effect on the surface electrical resistance and UV protection properties. The surface electrical resistance decreased by about 1.94 times with the increase in basis weight from 40 to 70 g/m². The lowest surface electrical resistance was obtained at the RGO coated cotton nonwoven fabric with the highest basis weight (70 g/m²). After the washing process, the surface electrical resistance of all RGO coated cotton nonwoven fabrics increased and the lowest value is obtained at the 70 g/m² basis weight nonwoven fabric with the percentage change in electrical resistance of 59.9%. Hydrophobicity was obtained with the reduction of GO. The UV transmittance of cotton spunlace nonwoven fabrics decreased prominently after the reduction of GO. Besides, UPF of cotton spunlace nonwoven fabrics increased with the reduction of GO and also, it is revealed that the UPF of RGO coated cotton nonwoven fabrics mainly affected by the basis weight of the initial cotton spunlace nonwoven fabric. The RGO coated cotton spunlace nonwoven fabric is a promising candidate with advantages such as low cost and easy processability for application areas such as wearable devices and smart textiles where flexibility, lightweight and functionality required.

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