POLİTEKNİK DERGİSİ

JOURNAL of POLYTECHNIC

ISSN: 1302-0900 (PRINT), ISSN: 2147-9429 (ONLINE)

URL: http://dergipark.org.tr/politeknik

Effect of alkaline hydrolysis process on the physical and electrical properties of reduced graphene oxide coated polyester knitted fabric

Alkali hidroliz işleminin indirgenmiş grafen oksit kaplı polyester örme kumaşın fiziksel ve elektriksel özelliklerine etkisi

Yazar(lar) (Author(s)): *Nergis DEMİREL GÜLTEKİN*[1]

ORCID[1]*: 0000-0002-1526-9382*

To cite to this article: Gültekin N. D., "Effect of alkaline hydrolysis process on the physical and electrical properties of reduced graphene oxide coated polyester knitted fabric", ***Journal of Polytechnic***, *(*): *, (*).

Bu makaleye şu şekilde atıfta bulunabilirsiniz: Gültekin N. D., "Effect of alkaline hydrolysis process on the physical and electrical properties of reduced graphene oxide coated polyester knitted fabric", ***Politeknik Dergisi***, *(*): *, (*).

Erişim linki (To link to this article): http://dergipark.org.tr/politeknik/archive

DOI: 10.2339/politeknik.1309440

Effect of Alkaline Hydrolysis Process on the Physical and Electrical Properties of Reduced Graphene Oxide Coated Polyester Knitted Fabric

Alkali Hidroliz İşleminin İndirgenmiş Grafen Oksit Kaplı Polyester Örme Kumaşın Fiziksel ve Elektriksel Özelliklerine Etkisi

Highlights

- *Polyester knitted fabric was treated with NaOH, coated with graphene oxide (GO), and then reduced with L-ascorbic acid.*
- *The surface morphology and chemical structure were characterized by SEM and FTIR, respectively.*
- *Abrasion resistance, bursting strength, electrical surface, and volume resistivities were investigated.*

Graphical Abstract

Polyester knitted fabric was treated with NaOH solution to impart surface roughness to the fabric. Then, untreated and alkali-treated fabrics were dip-coated with GO aqueous dispersion.

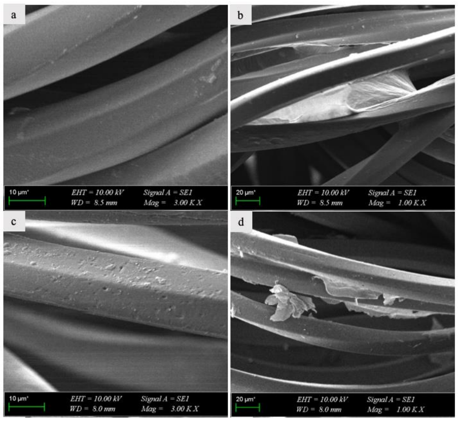


Figure. SEM images of (a and c) untreated and alkali-treated polyester fabric; (b and d) GO-coated untreated and alkali-treated polyester fabric.

Aim

Performing alkaline hydrolysis on polyester fabric and evaluating the effects on physical and electrical properties of GO and reduced graphene oxide (RGO)-coated polyester fabric.

Design & Methodology

Surface roughness was formed by alkaline hydrolysis treatment on polyester fibers, and dip coating of GO dispersion was applied followed by the reduction process.

Originality

The effects of alkaline hydrolysis treatment of polyester on the coating of GO, and also on physical and electrical conductivity properties of RGO-coated samples were investigated.

Findings

The electrical surface and volume resistivities of alkali-treated RGO-coated polyester fabric were obtained as 6.86×10^{4} *Ω/sq and* 1.51×10^{5} *Ωcm, respectively.*

Conclusion

The electrical surface and volume resistivities of alkali-treated RGO-coated polyester fabric were obtained as the lowest. The bursting strength of the alkali-treated polyester knitted fabric samples was obtained lower than the untreated samples. The polyester fabric and GO-coated polyester fabric showed increased abrasion resistance after the alkali treatment.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Effect of Alkaline Hydrolysis Process on the Physical and Electrical Properties of Reduced Graphene Oxide Coated Polyester Knitted Fabric

(This study was presented at ITFC 2023 conference.)

Research Article

Nergis DEMİREL GÜLTEKİN[1*]

[1]Faculty of Technology, Department of Textile Engineering, Marmara University, Türkiye

(Geliş/Received : 03.06.2023 ; Kabul/Accepted : 16.08.2024 ; Erken Görünüm/Early View : 12.09.2024)

ABSTRACT

In this research, the influence of surface treatment of polyester weft knitted fabric via alkaline hydrolysis on graphene oxide coating and reduction process was analyzed. In this regard, sodium hydroxide solution was prepared and the chemical etching of polyester fabric was carried out to create a surface roughening effect. Then, untreated and sodium hydroxide-treated fabrics were dip-coated with graphene oxide aqueous dispersion followed by a reduction process with L-ascorbic acid (Vitamin C) known as green reductant. The weight changes after each treatment were calculated. The changes in surface morphology and chemical structure of fabric samples were examined. The electrical resistivity of the fabric samples was tested using a setup consisting of a sourcemeter and resistivity test fixture. The physical properties of polyester knitted fabric were determined by employing abrasion resistance and bursting strength before and after alkaline hydrolysis, graphene oxide coating, and reducing processes.

Keywords: **Alkaline hydrolysis, polyester fabric, graphene oxide, dip coating, ascorbic acid reduction.**

Alkali Hidroliz İşleminin İndirgenmiş Grafen Oksit Kaplı Polyester Örme Kumaşın Fiziksel ve Elektriksel Özelliklerine Etkisi

ÖZ

Bu çalışmada, alkali hidroliz işlemi ile yüzey modifikasyonu uygulanan poliester atkılı örme kumaşın grafen oksit ile kaplanması ve indirgenmesi incelenmiştir. Bu kapsamda, poliester örme kumaş öncelikle sodyum hidroksit çözeltisi ile işlem görerek kumaşta yüzey pürüzlülüğü elde edilmiştir. Ardından, işlem görmemiş ve sodyum hidroksit ile işlem görmüş kumaşlar grafen oksit sulu dispersiyonu ile daldırma yöntemiyle kaplanmış ve sonrasında doğal bir indirgeme maddesi olan L-askorbik asit (C Vitamini) ile indirgenme işlemine tabi tutulmuştur. Her bir işlemden sonra kumaştaki ağırlık değişimi hesaplanmıştır. Poliester örme kumaşın fiziksel özellikleri, alkali hidroliz uygulanmış ve uygulanmamış numunelerde grafen oksit kaplama ve indirgeme işlemleri öncesi ve sonrasında aşınma dayanımı ve patlama mukavemeti ile belirlenmiştir. Taramalı elektron mikroskopu ve Fourier dönüşümlü kızılötesi spektroskopisi kumaş numunelerinin yüzey ve kimyasal yapısının incelenmesinde kullanılmıştır. Kumaş numunelerinin elektriksel yüzey ve hacim direnci bir kaynakmetre ve direnç test fikstüründen oluşan düzenek ile test edilmiştir.

Anahtar Kelimeler: Alkali hidroliz, polyester kumaş, grafen oksit, kaplama, askorbik asit ile indirgeme

1. INTRODUCTION

Textile materials have been gaining increasing attention in technical end-use areas beyond traditional applications [1]. Textiles offer various advantages, such as good flexibility and mechanical properties, high surface area, and lightweight. These characteristics make textiles ideal supports for producing functional materials [2, 3]. Various approaches, such as spinning, weaving, knitting, printing, and coating can be utilized to incorporate functionalities into textiles [4]. Among the several approaches that developed to produce functional textiles, the coating of textiles with nano-sized materials is considered one of the most effective ones [5].

Graphene oxide (GO) sheets, with their hydrophilic groups, can be dispersed in water, allowing for the deposition of graphene oxide onto textile materials through simple coating methods. Subsequently, GO can be converted to reduced graphene oxide (RGO), which is the electrically conductive form, using a chemical reduction method with a reducing agent [6, 7].

Polyethylene terephthalate (PET) fiber has various application areas, such as sports, apparel, activewear, medical textiles, protective clothing, and technical applications [8, 9]. Mechanical properties, durability, and abrasion resistance are major characteristics of polyester fibers [10]. However, the low hydrophilicity of polyester fabrics causes problems in applications such as finishing,

**Corresponding Author*

e-mail : ndemirel@marmara.edu.tr

dyeing, and washing. The surface modification of polyester fabric is generally applied to improve physical and chemical properties [11]. Alkaline hydrolysis is an effective method for introducing roughness to the fiber surface. The mechanism of the alkaline hydrolysis is based on the nucleophilic attack of hydroxide ions on the carbonyl group of polyester chains, which causes to breakage of the ester linkages, and thus, hydroxyl and carboxylate end groups occur. As a result of the alkaline hydrolysis process, physical and chemical effects occur in the fiber surface through chemical and morphological changes, resulting in the formation of small craters and functional groups. Thus, the fabric has better wettability [11, 12]. Sodium hydroxide (NaOH) is the most active and inexpensive material used for surface modification of polyester [13]. Several studies have been suggested about the surface modification of polyester to enhance the coating efficiency of nanoparticles such as TiO_2, carbon black and carbon nanotubes [14-16], nano-chitosan [17], silver nanoparticles [18], nickel nanoparticles [19], SiO_2 nanoparticles [20]. Kale et al. [21] studied the electrical conductivity, UV protection, and mechanical properties of alkali-treated polyester woven fabric with GO coating via simultaneous reduction using $TiCl_3$ aqueous solution. As a result, it is indicated that graphene/TiO_2 nanocomposite-coated polyester fabric showed excellent mechanical, UV protection, and electrical conductivity properties. Xue et al. [22] studied the graphene-ammonium polyphosphate (APP) composite aerogel coating on alkali-treated polyester fabric. The results showed that graphene aerogel-APP nanocomposite improved the flame retardancy of polyester fabric. Moazami et al. [23] studied the antibacterial activity and electrical resistivity properties of alkali-treated or ethylene diamine-modified reduced graphene oxide/Ag nanoparticles-coated polyester woven fabric. The effect of surface modification methods was examined and the aminolyzed polyester fabric exhibited lower electrical resistivity while higher antibacterial properties were obtained with the hydrolyzed polyester fabric.

In this regard, the study aims to perform alkaline hydrolysis at moderate conditions on polyester weft knitted fabric and then evaluate the effects on the physical and electrical properties of GO and RGO-coated polyester fabric. The alkaline hydrolysis was performed on the polyester weft knitted fabric with sodium hydroxide solution to impart nano-scale roughness. The process conditions were chosen as 70 °C and 30 min to consider the low energy consumption. Then, untreated and alkali-treated fabrics were dip-coated with graphene oxide aqueous dispersion followed by a reduction process with L-ascorbic acid (Vitamin C) known as green reductant. The physical properties of polyester knitted fabric were determined by employing abrasion resistance and bursting strength before and after alkaline hydrolysis, graphene oxide coating, and reducing processes. The electrical resistivities of the fabric samples were also tested using a setup consisting of a sourcemeter and resistivity test fixture.

2. MATERIAL and METHOD

2.1. Materials

Polyester weft knitted fabric (PF) (1x1 rib, 8 wale/cm, 16 course/cm, 182 dtex f 35) with a basis weight of 300 g/m^2 was used as the substrate. The chemicals for the experimental study were used as received without further purification. Flake graphite and L-ascorbic acid were obtained from Sigma Aldrich. Sulfuric acid (H_2SO_4, 95-98%), potassium permanganate ($KMnO_4$), and ethanol (C_2H_6O) were purchased from Isolab. Hydrochloric acid (HCl, 37%), phosphoric acid (H_3PO_4), sodium hydroxide (NaOH), and hydrogen peroxide (H_2O_2, 35%) were purchased from Merck. A nonionic washing agent (Perlavin OSV, Textilchemie Dr. Petry GMBH, Germany) was used to remove impurities on the polyester fabric.

2.2. Synthesis of Graphene Oxide

Graphene oxide was synthesized through improved Hummer's method [24]. Firstly, graphite flakes (3 g) were put into a beaker and stirred with a mixture of H_2SO_4/H_3PO_4 (360:40 mL). Then, 18 g of $KMnO_4$ was slowly added to the mixture. After the dissolution of potassium permanganate was completed, the reaction was placed in an oil bath, heated to 50 °C and stirred for 12 h. Afterward, the reaction was let to cool to 25±2°C and followed by pouring it onto 400 mL of ice. By the addition of 6 mL of 35% H_2O_2, the reaction terminated. Finally, the purification step took place by successive centrifugation with 1 M HCl and ethanol (2×). The washing process was applied through centrifugation until a pH of 4-5 was achieved. To obtain solid GO nanosheets, the as-obtained product was dried at 60°C.

2.3. Alkaline Hydrolysis of Polyester Knitted Fabric

A nonionic washing agent (Perlavin OSV) was used to remove impurities of polyester fabric. The fabric was treated with the solution with a bath ratio of 15:1 for about 30 min at 60°C, followed by rinsing with excess water and room-temperature drying. The alkaline hydrolysis treatment was applied by immersing the polyester knitted fabric in a NaOH solution (8%) with an L:G (liquor to goods) of 20:1 at 70°C for about half an hour. The treated fabric was neutralized by using 1%wt acetic acid solution followed by rinsing with excess distilled water. The NaOH-treated polyester fabric (PFN) was dried at 95°C for 60 min.

2.4. Coating of Polyester Knitted Fabric with GO

The 2 g/L aqueous GO dispersion was prepared using an ultrasonic bath. Polyester knitted fabric and NaOH-treated fabric were then immersed in the GO dispersion for 30 min at 60°C. Afterward, the GO-coated polyester fabric (PF-GO) and NaOH-treated GO-coated polyester fabric (PFN-GO) were put in the oven at 70°C for 60 min.

The dip-coating application was repeated 5 times to obtain a higher amount of GO nanosheets on the fabric.

2.5. Reduction of GO Coated on Polyester Knitted Fabric

The GO-coated and NaOH-treated GO-coated polyester fabric were chemically reduced using an aqueous solution of L-ascorbic acid. The reduction solution with a concentration of 0.2 M was prepared by dissolving L-ascorbic acid in distilled water and the bath ratio was kept at 20:1. Fabrics were immersed in L-ascorbic acid solution for 45 min at 95°C. The resulting RGO-coated fabric (PF-RGO) and NaOH-treated RGO-coated fabric (PFN-RGO) were rinsed with distilled water, followed by a drying process in an oven at 90°C.

2.6. Characterization

The Fourier transform infrared spectrometer with an attenuated total reflection (ATR-FTIR, Perkin Elmer Spectrum 100) was employed to characterize the chemical structure of fabric samples. Scanning electron microscopy (SEM, Zeiss EVO MA10) was used to analyze the surface properties of fabric samples. The polyester fabric samples were coated before the analysis with Au/Pd (Quarum SC7620) for about 120 sec.
The weight loss of the fabrics after NaOH treatment and the weight increase due to GO coating were calculated using the following equations:

$$\text{Weight loss } (\%) = \left(\frac{W_0 - W_1}{W_0}\right) \times 100 \quad (1)$$

$$Weight\ increase\ (\%) = \left(\frac{W_f - W_i}{W_i}\right) \times 100 \quad (2)$$

where W_0 is the weight of the untreated polyester fabric, W_1 is the weight of the polyester fabric after alkaline hydrolysis treatment; W_f is the final weight of polyester fabric after the GO coating process, W_i is the initial weight of polyester fabric before the GO coating process.
The mechanical property of the untreated and alkali-treated polyester knitted fabric, GO and RGO coated polyester knitted fabrics were tested by employing bursting strength and distension using m229 AutoBurst (SDL ATLAS) according to the TS 393 EN ISO 13938-1 standard at 10 cm^2 test area. Each fabric sample was tested three times and the average value was used after the diaphragm correction. The surface and volume resistivity of the fabric samples were tested using a setup consisting of a sourcemeter (Keithley 2450 Sourcemeter) and a resistivity test fixture (Keithley 8009). To measure the surface resistivity, a voltage potential across the surface of the specimen is applied, whereas volume resistivity is measured by applying a voltage across the sample. The following equations were used to calculate the surface and volume resistivities of the samples:

$$\rho_s = \frac{53.4 \times V}{\mathrm{I}}\ ohm\ (\Omega) \quad (3)$$

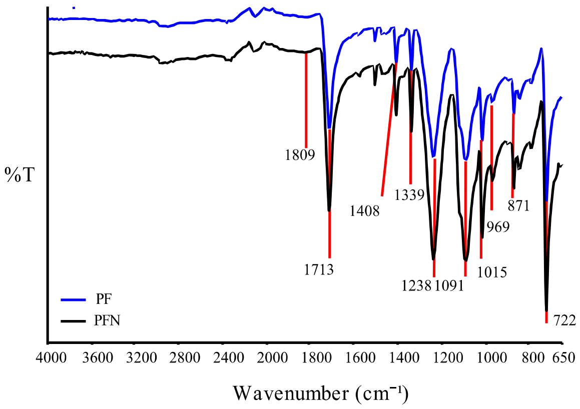
$$\rho_v = \frac{22.9 \times V}{\mathrm{t} \times \mathrm{I}}\ ohm.cm\ (\Omega.\mathrm{cm}) \quad (4)$$

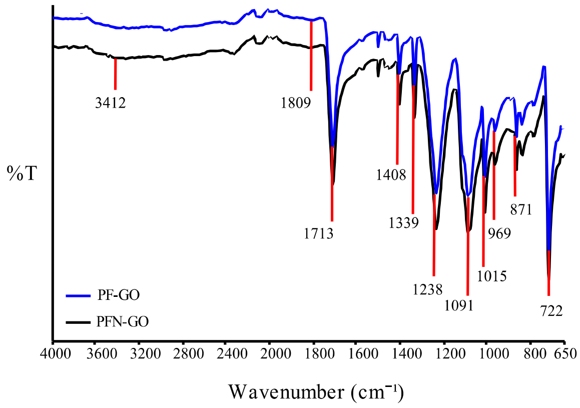
where ρ_s and ρ_v are the surface and volume resistivity of the specimen, respectively, V is the voltage, I is the current, and t is the average thickness of the specimen in cm. The abrasion resistance test was applied by using a Martindale pilling and abrasion instrument according to TS EN ISO 12947-2 standard. As an abrasive element, a standard wool woven fabric was used and 9 kPa pressure was applied to samples. The test is terminated when the breakdown of the fabric occurs.

3. RESULTS

3.1. FTIR Analysis

The effect of alkaline surface treatment on the PF and the effect of GO coating and reduction process were analyzed by FTIR. The FTIR analysis of untreated and alkali-treated polyester knitted fabric is given in Figure 1(a). The C–H bond in the aromatic group was found at 722 cm^{-1}, 871 cm^{-1}, and 1408 cm^{-1}, whereas C=O and C-O stretching vibrations of aromatic ester and ester appeared at 1713 cm^{-1} and 1238 cm^{-1}, respectively [25, 26]. The peak at 1015 cm^{-1} was assigned to the C–O stretching of glycol, and benzene in-plane vibrations appeared at 969 cm^{-1}. The peak at 1091 cm^{-1} corresponded to the ester C=O stretching vibration [3]. With the alkali treatment, the intensity of the peaks at 1713 cm^{-1}, 1238 cm^{-1}, 1091 cm^{-1}, and 722 cm^{-1} increased. Figure 1b, between 3600-2800 cm^{-1} a wide peak was observed which corresponds to O–H stretching vibrations of carboxylic acid. The abundance of alcohol groups and water molecules in GO could be the reason for the formation of this broad peak [27]. In Figure 1c, when the RGO was obtained, the broad peak between 3600-2800 cm^{-1} disappeared, and also, the decrease in the intensity of the sharp peaks was observed.





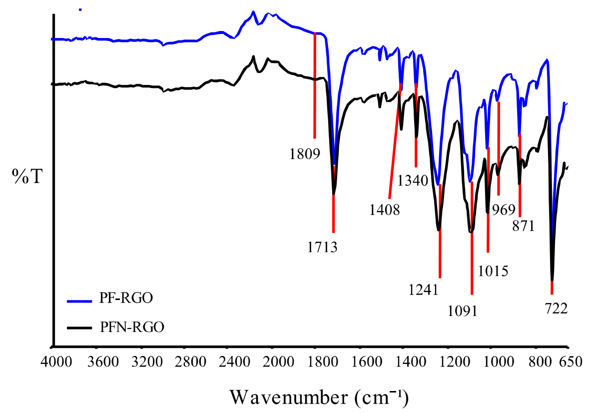


Figure 1. FTIR spectra of before and after NaOH-treatment (a) polyester fabric, (b) GO-coated, and (c) RGO-coated fabric samples.

3.2. SEM Analysis

Surface characteristics of the untreated and alkali-treated polyester fabric before and after the GO coating process are illustrated in Figure 2. The SEM micrograph of the PF demonstrates that the polyester filaments have a smooth surface. With the alkaline hydrolysis treatment, the surface morphology of the polyester filaments has changed with the formation of nano-sized pits and pores. This has confirmed that alkaline hydrolysis takes place on the surface of the polyester filaments. In Figures 2b and 2d, the presence of GO nanosheets can also be seen between and around the filaments which confirms that the coating process of GO nanosheets from the aqueous dispersion was successfully applied.

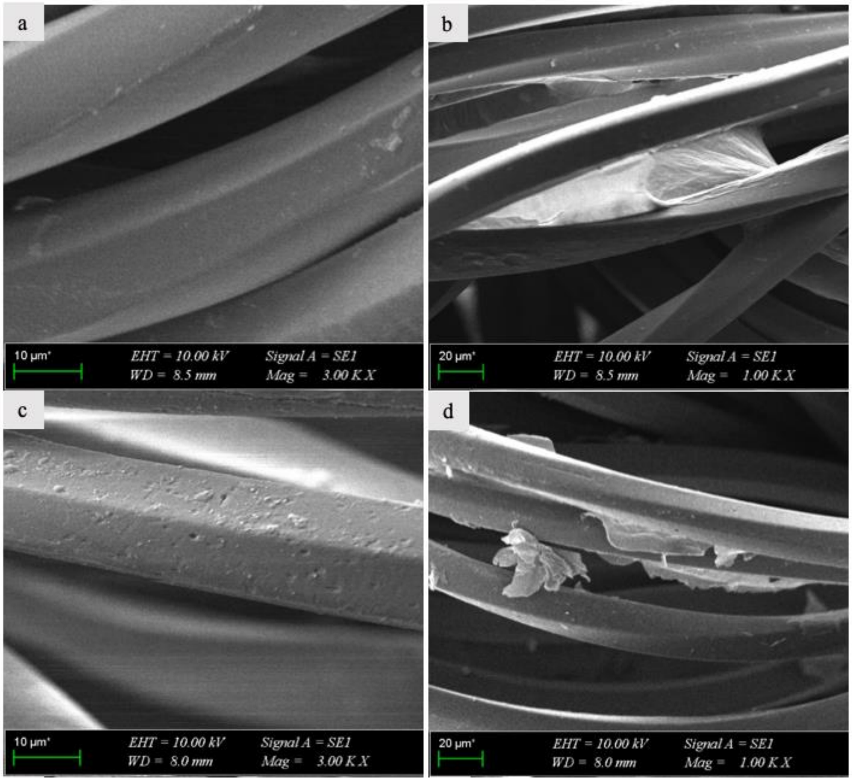


Figure 2. SEM micrographs of PF and PFN samples (a and c), PF-GO and PFN-GO samples (b and d).

3.3. Weight Changes

The process parameters of the polyester knitted fabric are given in Table 1. The weight loss after alkali treatment and the weight increase after the GO coating process of polyester knitted fabric can be found in Table 1. The alkaline hydrolysis is a well-known process as the weight reduction technique [8]. The weight loss of PF after alkali treatment is obtained as 4.48%. It is noteworthy that a moderate amount of NaOH was used and a lower temperature was applied for the surface treatment of polyester fabric compared to the other studies in the literature [12, 28]. According to this, it is reasonable to obtain lower weight loss after alkali treatment. The weight increase of the PFN-GO likewise the weight loss after the reduction of GO is obtained higher than that of the PF. The weight increase of PFN-GO can be explained by the formation of new active sides, mostly consisting of carboxylic acid groups on the polyester filaments by the alkaline hydrolysis process [29]. Thus, the more active sides of alkali-treated polyester filaments increase the physical adsorption of GO nanosheets. Besides, the surface roughness of the polyester filaments was obtained due to the chemical etching process applied by alkaline treatment. Therefore, with the increase in wettability of polyester fabric after alkaline hydrolysis, a higher amount of GO nanosheets was coated on the fabric [13]. Tavanai and Kaynak [30] indicated that the weight percentage of polypyrrole (PPy) on the polyester fabric increased with alkali hydrolysis treatment.

3.4. Electrical Resistivity

The surface and volume electrical resistivities of untreated and alkali-treated polyester fabric, after GO coating and GO reduction process are given in Figure 3. In Figure 3a, it is clear that the high electrical resistivity values of PF and PFN samples indicate that they are electrically insulators. Furthermore, the highly oxygenated functionalities of GO, make it also an electrical insulator [24]. For this reason, the electrical surface and volume resistivities of PF-GO and PFN-GO samples were measured in the range of 10^9 Ω/sq and Ωcm and 10^8 Ω/sq and Ωcm, respectively (Figure 3b). After applying the reduction process with L-ascorbic acid, a sharp decrease in surface and volume resistivities of samples was obtained. After the reduction process, the surface and volume resistivities of untreated and alkali-treated polyester fabrics gave different results. As can be seen in Figure 3b, the resistivity results of alkali-treated RGO-coated polyester fabric indicate the lowest values. Also, as indicated in Table 1, a higher weight increase was obtained after the GO coating of alkali-treated polyester fabric, as a result of successful surface etching and the creation of more active sides due to the alkali treatment. The surface and volume resistivities of PF-RGO and PFN-RGO were obtained as 1.30×10^5 Ω/sq and 2.94×10^5 Ωcm, 6.86×10^4 Ω/sq and 1.51×10^5 Ωcm, respectively. The decrease in both surface and volume resistivity is the result of the higher amount of RGO nanosheet deposited on the surface of alkali-treated polyester fabric [31]. Yıldız et al. [32], reported that the increased weight uptake and decreased surface electrical resistivity of polypyrrole (PPy)-coated polyester fabric was obtained with the alkali-treatment process. Aizamddin and Mahat [33] also reported that the polyaniline (PANI)-coated polyester fabric showed the highest electrical conductivity after the alkali hydrolysis process.

Table 1. The process parameters of polyester knitted fabric and corresponding sample codes.

Sample Codes	NaOH (%) (owf)	Weight loss[1] (%)	Sample Codes	GO[a] (g/L)	Weight increase[2] (%)	Sample Codes	L-ascorbic acid[b] (M)	Weight loss[3] (%)
PF	-	-	**PF-GO**	2	1.59	**PF-RGO**	0.2	0.66
PFN	8	4.48	**PFN-GO**	2	2.90	**PFN-RGO**	0.2	1.22

1: Weight loss of polyester knitted fabric after NaOH treatment. 2: Weight increase of polyester knitted fabric after the GO coating process. 3: Weight loss of RGO-coated polyester knitted fabric after the reduction process of GO. a: GO aqueous dispersion concentration. b: L-ascorbic acid solution concentration.

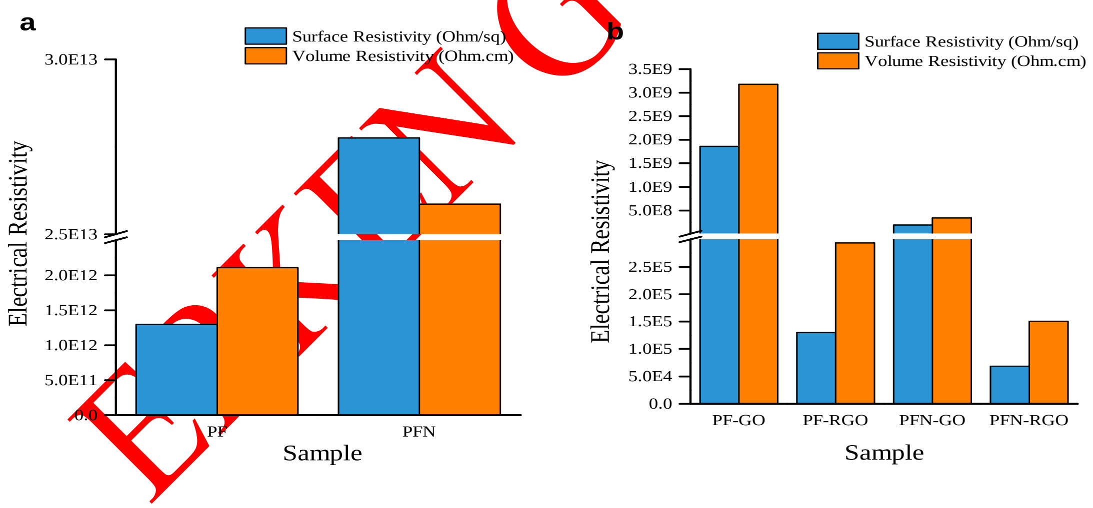


Figure 3. Surface and volume resistivities of PF and PFN samples (a), and GO and RGO coated untreated and NaOH-treated samples (b).

3.5. Mechanical Property

Figure 4 shows the bursting strength and bursting distension of untreated and alkali-treated GO and RGO-coated polyester knitted fabrics. The bursting strength of the alkali-treated polyester fabric samples was obtained lower than the untreated samples. The decrease in mechanical properties after the alkaline hydrolysis process is an expected result because of the chemical etching and formation of damages on the filaments [34]. However, the bursting strength of PFN increased after GO coating (PFN-GO) while the bursting strength of the PF decreased after GO coating (PF-GO). Figure 4b indicates the bursting distension of polyester knitted fabric samples was not affected by the alkali treatment.

The abrasion resistance of polyester fabric samples is shown in Figure 5. The PF sample showed the lowest abrasion resistance which was then increased with GO coating and further increased with the reduction of GO. This result can be explained by the molecular lamellar arrangement of the GO nanosheets acts as a self-lubricant

[35]. The NaOH treatment increased the abrasion resistance of the PF and PF-GO fabric. The alkaline hydrolysis process enhances the polyester filament surface more resistant to abrasion damage. The formation of microscopic damage on the surface of the polyester filaments causes less friction between them during the test, thus increasing the abrasion resistance [36].

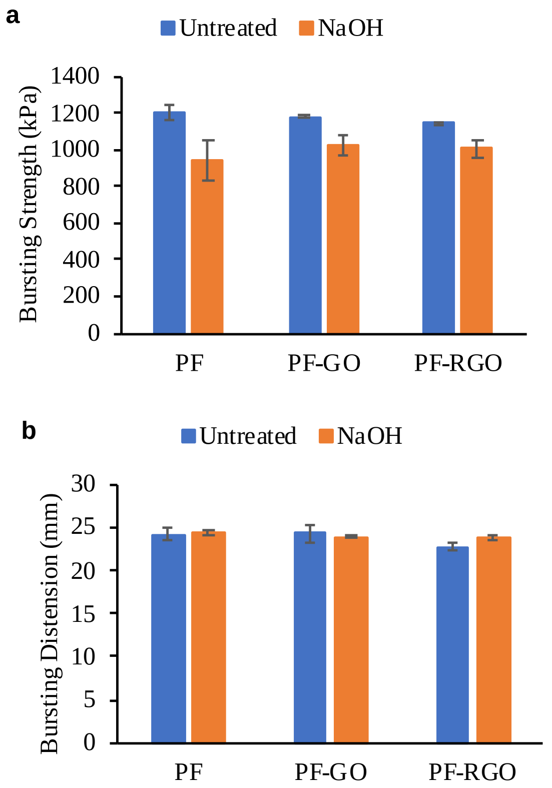


Figure 4. Bursting strength (a) and bursting distension (b) of untreated and NaOH-treated samples.

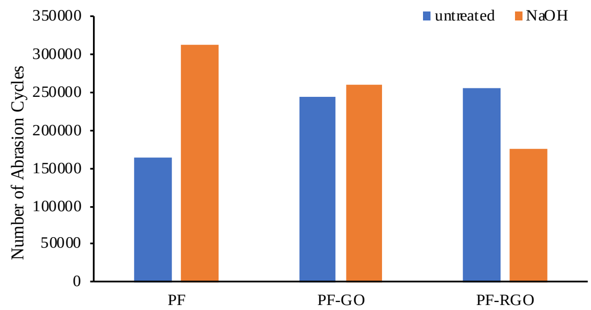


Figure 5. Abrasion resistance of untreated and NaOH-treated polyester fabrics, GO and RGO coated untreated and NaOH-treated samples.

4. CONCLUSION

The influence of surface treatment of polyester weft knitted fabric via alkaline hydrolysis on graphene oxide coating and reduction process is investigated. The successful application of alkaline hydrolysis treatment has been revealed from FTIR with the increase in intensity of carboxyl and hydroxyl groups, and from SEM with the pores on the surface of filaments. The weight loss of polyester fabric was obtained after the alkaline hydrolysis. Moreover, the percentage of weight increase of GO-coated polyester fabric was found higher than GO-coated untreated polyester fabric. The alkaline hydrolysis process has decreased the electrical resistivity of RGO-coated polyester fabric. The mechanical properties of polyester knitted fabric decreased by the alkaline hydrolysis treatment. However, the alkaline hydrolysis treatment did not effect the bursting distension of polyester fabric samples.

DECLARATION OF ETHICAL STANDARDS

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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