A Novel Method For The Fibre-Based Conductive Ring Spun Yarn Production

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Abstract

To take the advantages of spun yarns such as porosity, softness, bending as well as usability as yarn/fabric forms, in this study, it was worked on an alternative conductive yarn production method. Different from other methods such as coating, core-spun, blending, a conductive nanosuspension was applied to viscose, cotton and polyester open fibre bundles with different feeding amounts during the ring spinning with a specially developed apparatus. Reduced graphene oxide (rGO) was used to impart conductivity. Different from literature, rGO was synthesized with a single step process instead of two-step processes to ensure simple, easy-to-apply process and industrial applicability. Following to yarn production, winding, knitting and washing processes were realized to evaluate the changes in yarn conductivity and the usability of the yarns in the post-spinning processes. In addition to tensile properties of the yarns and air permeability of the fabrics, electrical resistance and environmental impact of the method was compared with immersion&drying process. The results indicated that alternative method allows the production of conductive (lower resistance than 100 kΩ) but also stronger, flexible, washable and breathable electronic textile products with an environmentally friendly process. There has been no effort, as yet, to get conductivity in this manner. Therefore, the developed method can be considered to be a new application in the functional yarn production field. The produced conductive yarns can be converted into fabric form by weaving, knitting and embroidery. Therefore, they can also be seen as an ideal as the platforms for future wearable electronics.

1. Introduction

Electronic textiles draw attention with its applications in health, defence and daily life due to these broad and functional features. Consumer demand for electronic textiles for personal use in daily life or sports activities shows that electronic textile market will grow even more in the coming period (Gonçalves et al. 2018). The biggest expectation in electronic textile market is the properties of flexibility, lightness, breathability, washability, and bending besides the high conductivity and electrical stability. In short, electronic textiles should be wearable. Another expectation is the production of electronic textile products with high quantity, low cost, easily applicable and environmentally friendly processes. Nowadays, most of the wearable textile products are still in the prototype stage and commercial examples are not so many. However, given the cost, performance and comfort, electronic products are still not met by consumers.

Today, conductive textiles are produced by methods such as: (i) using conductive metallic wires, (ii) adding conductive materials during the fiber drawing stage, (iii) coating fibers, yarns and fabrics with conductive materials, and (iv) coating conductive polymers on textile surfaces. Organic and inorganic materials such as metal wires, metal oxides, metal salts, conductive polymers, conductive ink, carbon nanotubes, carbon black and graphene-based nanoparticles are used to provide conductivity to textile fabrics. Despite providing conductivity to fabrics with the specified methods and materials, the major disadvantages are known to be harsh handle, increased weight, causing a rigid structure, loss of permeability and mechanical properties (Xue et al. 2005; Lam Po Tang and Stylios 2006; Bedeloğlu et al. 2010; Guo et al 2012; Kadoğlu and Duran 2012; Shateri-Khalilabad et al. 2013a, 2013b; Zeng et al. 2014;

Liu et al. 2015; Wu et al. 2016; Ghahremani Honarvar and Latifi 2017; Quadil et al. 2017; Wang et al. 2017). In addition, it is necessary to consider the ease of application, cost and environmental effects of the methods.

Due to the mentioned problems, there is a need for production methods and materials that allow the production of flexible, lightweight and wearable electronic textiles. In this context, present study focused on an alternative production method based on imparting the electrical conductivity to the open fibre bundle before yarn and fabric production. As is known, compared to two-dimensional textile structures such as fabric, one-dimensional yarns have unique properties such as softness, lightness, elasticity, breathability, bending, deformability, washability and resistance to mechanical deformations. In addition, yarns can be used as a yarn form and also easily integrated into fabric structures by weaving, knitting and embroidering. To take these advantages of the yarns, present study aimed to make the fibre-based structure conductive and then to spun the yarn from these conductive fibres. In the study, conventional ring spinning method was used to spin the staple fibres into the yarn. One of the reasons for the usage of conventional ring spinning is that porous, breathable, bulky, flexible and bendable structures of the spun yarns are thought to be beneficial in enhancing the expected properties from electronic textiles.

For the conductive yarn production with an alternative method, at first, a suspension was prepared with a conductive material and then applied to the open fibre bundle during ring spinning with a specially developed apparatus. Basic principle of the alternative method used in the study is different from widely used conductive yarn production methods such as coating, core-spun yarn production, and blending with conductive fibres etc., and all the efforts of the method is based on making each of the fibres conductive. Imparting conductivity in fibre form by this method can contribute to maintain the porous and flexible structure of textile materials. In addition to electrical conductivity property, the applied method will enable the conventional ring spinning system to product the functional yarns with various features depending on the used material. The produced conductive or other functional yarns can be converted into fabric form by the methods such as weaving and knitting, or they can be integrated into the fabric in embroidery form.

In this study, graphene was used to gain the conductivity to the staple fibres. Graphene-based nanosuspension was prepared and applied to staple fibres. Due to excellent properties of graphene, today, combining graphene with textile fibers has inspired extensive interest for the specific applications such as sensors, flexible fibre-type actuators, robots, motors, photovoltaic cells and supercapacitors (Cheng et al. 2014). Wet-spinning method has been widely used for the graphene-based fibre or yarn production (Xu and Gao 2015). In wet-spinning, coagulation baths were prepared depending on the solvents of GO LC (graphene oxide liquid crystal) dopes. The stretching operation was applied to achieve high alignment of GO sheets and to help the formation of compact structures (Xiang et al. 2013; Xu et al. 2013). To obtain the highly compact structure of the final graphene fiber, chemical (Cong et al. 2012; Xu and Gao 2015) or thermal (Jalili et al. 2013; Aboutalebi et al. 2014) reduction was applied. Meng et al. (2013) worked on the production of a unique all-graphene core–sheath fibre and a core of GF was covered with a sheath of 3D porous network-like graphene framework. Xiang et al. (2013) used large flake

GO (22 μm average diameter) as the building blocks to improve the low tensile modulus of wet-spun GO fibres. Zhao et al. (2013) modified the wet-spinning process based on the direct spinning of hollow GO fibers in a coagulation bath of methanol solution, and used a coaxial two-capillary spinning principle for the production of controlled graphene-based hollow fibres. Aboutalebi et al. (2014) obtained GO fibres by wet spinning method using $CaCl_2$ and NaOH coagulation baths, and reduced by overnight annealing at 220°C under vacuum. Yun et al. (2015) demonstrated the effectiveness of wearable gas sensors based on reduced graphene oxide-decorated yarn (RGOY) with ultra-sensitivity at room temperature using a robust fibre wrapping method. Zhao et al. (2015) fabricated a novel all graphene coaxial fibre supercapacitor (GCS) consisting of a continuously wet-spun core graphene fibre and facilely dip-coated graphene sheath. Yu et al. (2017) investigated a wire-shaped supercapacitor with graphene fibre electrodes fabricated from chemical vapor deposition (CVD) grown laminated graphene film. As can be seen from the brief literature summary given above, graphene-based fibre or yarn production is mostly focused on the wet-spinning, which is one of the synthetic fibre spinning method. Therefore, the method used within the scope of the study, it is the first time in the literature that the fibre structures gain conductivity by applying graphene-based nanosuspension. In our previous study, conductive yarns were produced with the alternative method using carbon black particles (Kayabaşı et al. 2020). Unlike the literature, with the developed alternative method, it is aimed to gain conductivity to spun yarns consisting of staple fibers such as viscose, cotton, polyester etc. by using graphene nanosuspensions synthesized by a one-step process.

2. Experimental Details

2.1. Materials

In the production of conductive yarns with the alternative method, a graphene-based material produced by a method consisting of a one-step process without the need of chemical reduction was used. Therefore, reduced graphene oxide (rGO) nanosuspensions were synthesized. For the preparation of reduced graphene oxide (rGO) nanosuspensions, graphite powders (<20 μm, 12.01 g/mol) used to synthesize of GO purchased from Sigma Aldrich Co. (Germany). The chemicals required to synthesize graphene oxide (GO) were obtained from H_2SO_4 (98%) and H_2O_2 (30%) from Sigma Aldrich Co., HCl (37%) Merck (Germany). $KMnO_4$ were purchased from Tekkim A.Ş. (Turkey). Hydrazine hydrate supplied form Merck was used in the reduction of GO. In order to prevent agglomeration of the produced rGO nanosuspensions in the water base fluid, poly (sodium 4-styrene sulfonate) (PSS) with hydrophilic and anionic polyelectrolyte properties was used. PSS (Mw˜70000 g/mol) was supplied by Sigma Aldrich Co.

Synthesis of Graphene Oxide (GO): In the synthesis of graphene oxide (GO), the improved Hummer's method, whose synthesis steps are shown schematically in Figure 1, was used. Firstly, graphite powder is converted into graphene oxide nanotripts by being exposed to 9: 1 ratio Sulphuric acid-Ortho Phosphoric acid mixture and heavy oxidation in permanganate environment. In this context, 1 g of powder graphite is added to a mixture of 27 mL of concentrated H_2SO_4 and 3 ml of H_3PO_4 and mixed in the ultrasonic bath

for 5 hours. Then, $KMnO_4$ is added gradually in the ratio of 1: 6 (Graphite: $KMnO_4$) and mixed in the ultrasonic bath for another 8 hours. Then, at a ratio of 1: 80 by volume H_2O_2: H_2O is added to the mixture. It is washed with 0.1 M HCl at least 4 times to remove the impurities in the solution and then with pure water to have the pH of the mixture 7.

Production of Graphene/Poly (Sodium 4-Styrenesulfonate) Suspension: Graphene/poly (sodium 4-styrene sulfonate) (PSS) suspension production steps are shown schematically in Figure 2. First, 1 g of graphene oxide is added into 1000 mL of deionized water and mixed 30 minutes with a 750 W power ultrasonic mixer (Sonics & Materials ING, USA). Then, PSS is added into the mixture as 1:4 GO: PSS and mixed with ultrasonic mixer for a further 30 minutes. Reduction is performed by adding hydrazine hydrate into the mixture (GO: hydrazine hydrate = 1: 3.5 mg/μ L) at 115 ° C under 1.5 bar pressure for 3 hours in an autoclave. Then, the reduced graphene oxide/PSS produced is washed with deionized water at least 4 times to completely remove the hydrazine from the hydrate and then dried at 50°C. The desired nanosuspension is produced by adding rGO/PSS in water at a concentration of 10 mg/mL, mixing with ultrasonic mixer for 1 hour.

2.2. Method

In this study, it was focused on the usage of an alternative method which is based on imparting the staple fibre bundle electrical conductivity before the yarn formation (Figure 3). Different from the widely used conductive yarn production methods such as coating, core-spun yarn production, and blending with conductive fibres etc., the principle of the method is to gain electrical conductivity not only to the outer surface but also the internal structure of the yarn. In the alternative method, besides imparting electrical conductivity property, it was also aimed to protect the elasticity, permeability and open structure of the yarn material and thus not to have a significant loss in the properties related to fabric comfort. In the study, yarn production was realized by conventional ring spinning machine (Rieter G10 model), which is widely used in the spun yarn production. As stated above, conventional ring spinning was preferred in order to help enhancing the expected properties from electronic textiles in terms of the porous, breathable, bulky, flexible and bendable structures of the spun yarns. The principle of the alternative method is based on application of conductive nanosuspension on to the open fibre bundle during ring spinning with a specially developed apparatus (Figure 3a). In the method, it is aimed to make each fibre conductive. In this way, it is thought that gaining conductivity in fibre form will contribute to maintaining the porous and flexible structure of textile materials.

As known, the working principle of the ring spinning machine is based on the principle of drawing the roving strand by passing through the drafting zone, twisting by spindle-ring-traveller and winding the spun yarn onto the cops. During the conventional ring spun yarn production, drafted fibre bundle is converted to the spun yarn after the twisting with spindle-ring-traveller of ring spinning machine. In the alternative method, a feeding system consisting of a feeding pump (1), syringe (2) and a needle (3) was used to apply rGO nanosuspension into the fiber bundle (Figure 3b). Firstly, rGO nanosuspension was

mixed by an ultrasonic mixer for 2 hours. Then, rGO nanosuspension was filled into the syringe (2) and syringe was placed in the feeding pump (1). The feed pump provided the transferring rGO nanosuspension to the needle (3) by a syringe at a given feeding rate. Thus, it was possible to work with a fixed amount of feeding. The needle allowed nanosuspension to be applied to the desired area of the fibre bundle. In the study, it was focused on applying the nanosuspension to the fibre bundle prior to spun yarn formation in order to apply the suspension to the inner structure as well as the outer surface of the yarn. For this reason, the application was preferred in the main drafting region so that the application can be carried out before yarn formation. The needle of syringe was positioned at the midpoint of the open form fibre bundle passing through the main drafting region, and nanosuspension was applied to all fibres. After the conductive solution was fed into the fibre bundle, twist was gained through the spindle-traveller-ring, as in conventional ring yarn production, and the resulting yarn was wound on to the cops. The produced yarns have been left to dry under normal room condition.

On the other hand, applications in different feeding rates (mL/h) were carried out to research the changes in electrical conductivity values. Resistance values of the yarns were measured and optimum feeding rate was determined. Ring spun yarns with Ne 16/1 yarn count were produced (Table 1). In the study, viscose fibres were used predominantly because of the widely usage in the textile industry and having a hydrophilic feature. In addition to viscose fibre, cotton and polyester fibre rovings were used and rGO doped ring spun yarns were produced to determine whether there are any limitations of the alternative production method or not. Fiber length was 38 mm and fiber fineness was 1.3 dtex for viscose and polyester fibres. For cotton fibres, fibre length was 28.62 mm, fiber fineness was 1.66 dtex (Mic 4.22) and fiber tenacity was 30.58 g/tex (Özen 2020).

Table 1
Yarn production parameters in conventional ring spinning system

Parameter	Value
Roving count	Ne 0,81
Yarn count	Ne 16/1
Total draft	21,5
Spindle speed (rpm)	5500
Twist (t/m)	524
Twist coefficient (αe)	3,4

2.3. Test and Analysis

After the rGO application to the viscose fibre bundles, yarn images were taken with the FEI QUANTA FEG 250 Scanning Electron Microscopy (SEM) at 10 kV in order to determine the morphological properties of the yarn structure. Electrical resistance of the yarn samples after rGO application, which express the electrical conductivity of the produced yarns, was determined by two-probe method based on AATCC Test Method of 84-2005. Due to the shorter measurement time and ease of application, the two-probe measurement method was preferred for measuring the electrical conductivity of all rGO applied yarn samples. Electrical resistance of rGO doped yarns was measured with the EXTECH EX520 Digital multimeter (Extech Instruments Corporation). 10 measurements were taken from each fabric sample and average resistance values were calculated. In the study, yarn strength and breaking elongation properties of rGO nanosuspension doped yarns were tested by Mesdan Lab tensile tester according to ASTM D 2256. Test length was 500 mm and the test speed was 5000 mm/min and 10 tests were carried out for rGO doped and undoped reference yarns.

After yarn production, the yarns were wound onto the bobbin with winding process and then knitted to determine the effect of friction occurred during winding and knitting processes on electrical conductivity of the rGO doped yarns. Additionally, in this part, it was also aimed to evaluate the suitability of produced conductive yarns for industrial processes. The winding process was carried out on the Oerlikon Schlafhorst winding machine at a high winding speed of 1000 m/min, and the yarns were converted into knitted fabric form using the flat knitting machine (Hong Qi Ma brand, DB14-F model). Electrical conductivity of the yarns was determined by the two-probe measurement method. Fabric density was 56 loop/cm^2 and fabric weight was 186 g/m^2. In addition to two-probe method, measurement of electrical conductivity of knitted fabrics was repeated with the four-probe technique (AATCC 76-2005 Test Method) to compare the resistance values with the literature.

Today, it is very important for electronic textiles to maintain their electrical conductivity after washing. In order to investigate the washability the rGO applied fabrics, knitted fabric samples were washed with the non-ionic detergent at 40°C according AATCC Test Method 61- 2006 test standard. Resistance values of washed samples were determined by four-probe method. On the other hand, air permeability property of the knitted fabrics were tested in Textest AG FX 3300 based on TS 391 EN ISO 9237 standard in order to determine the properties of knitted fabrics related to fabric comfort. As is known, impregnation method is widely preferred to gain functional properties to the fabrics due to its low cost, easy application and does not require complex equipment. In order to compare the performance of the alternative method with the impregnation method, rGO nanosuspension was applied to the fabrics knitted from undoped reference viscose ring spun yarns using ATAÇ-FY350 laboratory foulard device. After application, rGO applied fabrics were dried at room temperature (about 25°C). Immersion&drying process was carried out 5 times and the electrical resistance values of the fabrics were determined by two-probe measurement method.

3. Results And Discussion

In this section, various properties of the yarns produced by the alternative method based on the application of a conductive nanosuspension to the open fibre bundle with an apparatus mounted on the

conventional ring spinning system consisting of a feeding pump, syringe and needle were examined. Additionally, some of the fabric properties knitted from these yarns are studied. The results were given below and ring spun yarn without rGO nanoparticles was named as a reference and undoped ring spun yarn while the yarn comprising rGO nanoparticles was called as a conductive and doped yarn.

3.1. Yarn Morphology

rGO nanosuspension was applied on to the viscose fibres at the 70 mL/h feeding rate and SEM images of Ne 16/1 undoped viscose ring spun yarns for different magnifications were analysed (Figure 4a). The images of rGO doped viscose yarns were compared with rGO undoped reference viscose ring spun yarns (Figure 4b).

As seen in Figure 4a, a clean structure could be seen between the fibres and also on the surface of undoped reference viscose ring spun yarn. Comparing with the images of untreated ring spun yarns, rGO nanoparticles were observed on the fibre surface (shown by red circles in Figure 4b). As observed in literature for fabric application (Molina et al. 2013, Shateri-Khalibad and Yazdanshenas, 2013a, 2013b; Karimi et al. 2014), rGO sheets wrinkled, folded and deposited on a fiber surface. According to yarn appearances given in Figure 5, white colour of viscose fibres were getting darker after the rGO nanosuspension application, and this case indicated the presence of rGO nanoparticles in yarn structure. In order to confirm this case, changes in the weight of reference (undoped) and rGO doped yarns were compared. It was found that the weight is 0.036016 g/m for undoped ring spun yarn while 0.038108 g/m for rGO doped yarn (at 70 mL/h). With the application of rGO nanoparticles, the weight of the rGO doped yarns increased. On the other hand, it was observed that reference and rGO applied yarns had similar yarn structure due to the real-twist given by ring-traveller during the classical ring spinning process. Both yarn types still carried the ring spun yarn character. However, it was determined that rGO applied ring spun yarns have lower number of protruding fibres and therefore compact yarn structure was observed on rGO doped yarns in comparison to undoped reference yarns.

Yarn Conductivity

In order to impart the electrical conductivity to viscose fibre bundle, different feeding rate values of reduced graphene oxide (rGO) nanosuspension were researched, and proper feeding rate value was determined. Feeding rates were increased from 30 mL/h to 80 mL/h and rGO nanosuspension was applied to viscose fibre bundle with the needle and syringe in a determined amount provided by the feeding pump. Electrical conductivity of the produced Ne 16/1 viscose ring spun yarns were measured by two-probe measurement method. During the experiments, no resistance value was determined at 30, 40 and 45 mL/h feeding rates, and therefore these feeding rates were concluded as insufficient. In addition, yarn colour was expected to change from white to black after the rGO nanosuspension application. However, it was observed the white or gray places on the yarn surface (shown by red circles in Figure 6) or yarn colour was not completely black. Thus, irregular colour distribution was determined through the yarn length due to insufficient rGO feeding. Following these feeding rates, yarn production was realized at

feeding rates of 50 mL/h and above, and electrical resistance value was detected in rGO doped viscose ring spun yarns. This case meant that produced yarns become conductive and show conductivity property after rGO nanosuspension application with the alternative application method. Electrical resistance results are given in Figure 7 for 50-80 mL/h. As seen, electrical resistance values decreased when the feeding rate was increased from 50 mL/h to 80 mL/h. At a feeding rate of 80 mL/h, it was determined that resistance values tend to increase. This finding indicated that rGO nanosuspension application with the alternative method improves the electrical conductivity up to a certain conductive solution feeding value. However, conductivity tended to increase above the limit rGO application value.

This case was similar to the results of the graphene application to textile fabrics in the literature. In the studies, woven and knitted fabrics were impregnated with GO and subsequently reduced by chemical or thermal reduction. In order to improve the electrical conductivity, application cycle comprising rGO nanosuspension padding and drying process was increased, and it was determined that the resistance values decreased up to a certain application cycles. In insufficient rGO nanosuspension application cycles, rGO layers may not be in contact so that electrical conduction is more difficult and higher resistance values were determined. When the rGO application was increased, resistance of the fabric samples tended to decrease due to the improved contact between the rGO sheets. Beyond certain application cycles, electrical resistance values started to increase slightly, and conductivity was getting lower. After a certain rGO deposition, the limit of conductivity was reached and more rGO did not produce an increase in the conductivity due to saturation of textile materials with rGO nanoparticles (Molina et al. 2013; Sahito et al. 2015).

On the other hand, as similar to our study, Karim et al. (2017) obtained GO with a modified Hummer's method and reduced GO chemically using a reducing agent ($Na_2S_2O_4$) to enable one-step graphene application process. rGO dispersion applied to the textile fabric using a simple pad-dry technique without the requirement of a reduction process. It was indicated that electrical resistance values reduce about ~90% after five application cycles. The authors explained the significant improvement in electrical conductivity of the fabrics by an absorption and adsorption phenomenon. In first few application cycles, absorption of rGO dispersion into the fibres was pre-dominant. The nanoplates could disperse on the fibre surface uniformly as the rGO application cycles were increased. When the saturation point was reached, rGO was then mainly adsorbed on the fibre surface and continuous conductive film was formed by creating better connections between flakes. Thus, the resistance of the fabric decreased by presenting more flakes on the fibre surface and by the restacking of the flakes through the Van der Waals forces applied by the squeeze rollers (Karim et al. 2017). As a result, electrical conductivity improved up to a certain rGO nanosuspension feeding rate as reported in rGO fabric application studies.

One the other hand, in the study, the lowest resistance value was determined as 1.008 MΩ/cm in rGO/viscose ring spun yarns at a feed rate of 70 mL/h produced with alternative method. The lowest variation in the resistance values and better stability of the rGO nanoparticles in the yarn structure was obtained at 70 mL/h and therefore 70 mL/h was decided as an optimum feeding rate (Figure 7).

Tensile Properties

In order to analyze the effect of rGO nanosuspension application on tensile properties of the yarns, tenacity and breaking elongation of rGO doped Ne 16/1 viscose ring spun yarns were analyzed. The results of the yarns were compared with undoped reference viscose ring spun yarns. As seen in Figure 8, tenacity of rGO doped yarns changed depending on rGO nanosuspension feeding rates. As the rGO feeding was increased, tenacity of rGO doped yarns was getting higher, and the highest yarn tenacity values were obtained at the highest rGO nanosuspension feeding rate values (75 mL/h and 80 mL/h). rGO doped viscose yarns had slightly higher yarn tenacity values at 75 mL/h and 80 mL/h feeding rates. The differences in tenacity values of doped (75 mL/h and 80 mL/h) and undoped yarns were about 2-3%. It was determined that optimum feeding rate of 70 mL/h ensure stronger and also conductive yarn production.

The increase in rGO doped yarn tenacity might result from reinforcing characteristic of graphene. Graphene has extraordinary mechanical properties, including a record tensile strength (130 GPa) (Xu and Gao 2015). In literature, it was reported that the addition of a low fraction of graphene nanosheets into polymer composites as a filler results in a significant improvement in the mechanical strength of the composites. It was stated that the carboxyl and hydroxyl functional groups on the basal planes and edges of the starting GO nanosheets may act as linkers between the graphene and the polymer (Cheng et al. 2014). Regarding graphene application to textile fabrics, it was indicated that the breaking and tearing strength of graphene (rGO) applied fabrics is increased (Yaghoubidoust et al. 2014; Gan et al. 2015; Karim et al. 2017). The presence of graphene particles had positive effect of mechanical properties and graphene was shown as the reason of this case (Gu and Zhao 2011; Abbas et al., 2013; Yaghoubidoust et al. 2014).

Breaking elongation of the yarns obtained with alternative and classical ring spinning methods was compared and the results are shown in Figure 9. As similar to yarn tenacity results, breaking elongation values of rGO nanosuspension doped yarns changed depending on nanosuspension feeding rates. According to the values, breaking elongation values of viscose ring spun yarns were about 15% while they were about 11.8-15% for doped yarns depending on feeding rates. rGO doped yarns produced at 70 mL/h feeding rate had similar yarn breaking elongation values with undoped reference viscose ring spun yarns. However, rGO doped yarns produced at lower and higher feeding rates than 70 mL/h had mostly lower elongation values than that of the undoped ring spun yarns. In literature, Kaynak et al. (1996) stated similar findings for carbon black (CB) containing composites and indicated that elongation at break values decreased sharply with the filler content for all types due to the stress–concentration effect. Therefore, breaking elongation values of rGO doped yarns might be decreased to some extent due to the incorporation of the rigid filler into yarn structure.

As a conclusion, yarn tenacity and breaking elongation results indicated that rGO nanosuspension applied yarns with the developed alternative method have comparable tensile properties with undoped reference viscose ring spun yarns. However, optimum feeding rate of nanosuspension was effective on

tenacity and breaking elongation values of the doped yarns. Particularly, insufficient nanosuspension feeding caused a loss of yarn tenacity and breaking elongation values of the yarns besides low electrical conductivity values.

Effect Of Fiber Type

In this part of the study, it was aimed to evaluate the performance of alternative production method using different fibre types. Therefore, in addition to viscose fibres, rGO nanosuspension was applied to cotton and polyester fibres. rGO nanosuspension was doped to hydrophilic (bleached) cotton and polyester fibre bundle with alternative method on conventional ring spinning machine at 70 mL/h feeding rate. Electrical resistance values of the ring spun yarns are given in Table 2.

As seen in Table 2, cotton and polyester fibres had a certain degree of electrical resistance and hence conductivity values. Therefore, developed alternative method allows the conductive yarn production with various fibre types. However, rGO doped cotton and polyester yarns had higher resistance than viscose ones. Hydrophobic structure of the cotton and polyester fibres was thought a possible for their lower conductivity values.

Table 2
Electrical resistance values of rGO doped ring spun yarns for different fibre types

Fibre type	Mean resistance (MΩ/cm)
Viscose	1.008
Cotton	21.780
Polyester	43.411

Effect Of Yarn Winding Process

In order to convert the rGO doped ring spun yarns into fabric form with industrial weaving and knitting processes, the yarns must be transferred from cops to bobbin in order to be used in post-spinning processes. For this reason, reference undoped and rGO applied viscose ring spun yarns were wound on to the bobbin by winding machine. As known, the yarn during the winding process passes from various parts of winding machine such as yarn guide, tension regulator, quality control units, and hence subjects to friction intensely. In order to evaluate the effect of friction on electrical conductivity of yarns, rGO doped ring spun viscose yarns were wound from cops to bobbin on the Schlafhorst winding machine at high production speeds of 1000 m/min (Figure 10). Three undoped and rGO doped yarn cops (produced at 70 mL/h feeding rate) were wound on the bobbin and ten electrical resistance measurements were done on the bobbin by two-probe measurement method. Electrical resistance values of the rGO yarn bobbins given in Table 3 indicated that the yarns still maintain their conductivity property after winding

process. However, the resistance values of the yarns increased approximately 2 times after the winding process. An expected increase might be resulted from friction occurred during the winding. In addition to the analysis of resistance results, the ratio of change in resistance values to initial resistance values was calculated ($\Delta R/R_0$). When the results were examined, it was determined that $\Delta R/R_0$ was very small (≈ 1).

Table 3
Electrical resistance results after winding process

Before winding (MΩ/cm)	After winding (MΩ/cm)	$\Delta R/R_0$
1.008	2.197	1.179

Conductivity Of Knitted Fabrics

In the study, rGO doped yarns were used in a flat knitting machine to investigate the usability of the yarns in the knitting process. In this part, it was also aimed to evaluate the changes in conductivity property of the rGO doped yarns against intense friction occurred in fabric knitting. The yarns rub against various metal parts such as thread guide, needle, and platinum during the knitting process. For the knitted fabric production, conductive viscose ring spun yarns wound on to the bobbin were used and electrical resistance values of the fabrics (Figure 11) were measured by two-probe method.

When the electrical resistance results were examined (Table 4), it was determined that the resistance values increase after fabric production, as expected. However, the ratio of change in resistance values to initial resistance value (from yarn cops to fabric) ($\Delta R/R_0$) was very small (<1) and the resistance values increased slightly. This result shows that electrical resistance values almost does not change during the processes from yarn to fabric production. Additionally, knitted fabrics obtained from rGO doped yarns had still electrical conductivity feature even after high friction effect in knitting process.

Table 4
Electrical conductivity results of fabric samples knitted from rGO doped viscose ring spun yarns

Resistance of yarn cops (MΩ/cm)	Resistance of yarn bobbin (MΩ/cm)	Resistance of knitted fabric (MΩ/cm)	Resistance change from cops to fabric form ($\Delta R/R_0$)
1.008	2.197	1.803	0.788

Comparison With Impregnation Method

To evaluate the performance of the alternative method, rGO nanosuspension was applied to the fabrics knitted from undoped reference viscose ring spun yarns. rGO nanosuspension was applied to 10 × 10 cm^2 knitted fabrics by immersion&drying method due to its low cost, easy application and does not

require complex equipment on the ATAÇ-FY350 laboratory type foulard device. After application, rGO doped fabrics were dried at room temperature. Immersion&drying process was repeated 5 times due to better conductivity values determined in literature (Javed et al. 2014; Sahito et al. 2015; Zhou et al. 2015) and also our previous study (Özen 2020; Özen et al. 2021). Resistance values of the fabrics were measured by the two-probe method. The results were compared with that of the knitted fabrics obtained from developed alternative application method. When the results were examined (Table 5), it was determined that knitted fabrics obtained from alternative application method have higher resistance and hence lower electrical conductivity values compared to the impregnation&drying method. However, the difference between resistance values of both application methods was about 4 times. Nevertheless, both fabrics had a resistance value of MΩ level and hence it is thought that the results of the fabrics are comparable. Higher resistance values obtained from alternative method indicated that amount of rGO nanoparticle applied with alternative method might be lower than that of the impregnation&drying method. Therefore, comparing with alternative application method, more connections between flakes realized by impregnation&drying method might lead to more reduction in resistance of the fabric.

Table 5
Electrical resistance results of the knitted fabric samples

Application type	Two-probe	Four-probe
Immersion&drying method (5 repeations)	0.456 MΩ/cm	-
Alternative method	1.803 MΩ/cm	1.64 kΩ.cm

In the study, electrical resistance of the knitted fabric samples was determined by four-probe method in order to compare the resistance values of this study with the findings given in literature. As seen in Table 6, the resistance values of the fabric samples knitted from rGO doped yarns were about 1.64 kΩ.cm and the values were lower than 100 kΩ. In literature, Fugetsu et al. (2010) classified the usage of conductive fabrics according to a resistance value of 100 kΩ.cm and stated that the fabrics with a resistance value of 100 kΩ.cm and lower values can be used in soft and flat electric heaters while the fabrics with a resistance value higher than 100 kΩ.cm can be used in antistatic products. Therefore, rGO applied fabric samples with a resistance value of 100 kΩ.cm could be thought as a considerably conductive material. On the other hand, an accurate and precise measurement of four-probe method led to a considerably difference (10^3) between the resistance values determined by two and four-point methods. Therefore, the results of both methods were not coincided with each other completely.

The Effect Of Washing On Electrical Conductivity

In order to investigate the washability the conductive rGO viscose knitted fabrics, fabric samples were washed with the detergent at 40°C. Resistance values of washed samples were determined by four-probe method and it was seen that electrical resistance values of rGO-containing fabrics tend to increase after

washing process (Table 6). However, the value was lower than 100 kΩ. Therefore, it can be said that laundering did not have considerable effect on the electrical conductivity of these fabric samples, and the samples kept their conductivity property.

Table 6
Electrical resistance results of the unwashed and washed fabric samples

Application method	Before washing (kΩ.cm)	After washing (kΩ.cm)	Rate of resistance change ($\Delta R/R_0$)
Alternative method (knitted fabric)	1.64	7.66	3.67

Air Permeability

Air permeability is an important factor used to provide an indication of the breathability of the fabrics and hence clothing comfort. In some products, high air permeability is desirable. In order to determine the effect of alternative application method on permeability property of the fabric, air permeability of knitted fabrics produced from rGO doped viscose ring spun yarns was compared with that of the reference fabrics obtained from undoped yarns. Test results were analyzed statistically by SPSS 16.0 statistical software to determine any significant differences. ANOVA multiple-range test (LSD method) were used and ANOVA analyses were performed for $\alpha = 0.05$ significance level (Table 8). According to Figure 12 and Table 8, it was observed that the fabrics produced from alternative application method had significantly higher air permeability values than the fabrics knitted from undoped yarns. In addition, all knitted fabrics produced at three different rGO feeding rates gave better air permeability values than undoped reference fabrics. This result indicated that knitted fabrics obtained from alternative application method have a more permeable structure resulted from compact structure of rGO doped viscose ring spun yarns. As seen in Figure 13, rGO doped yarns had lower protruding hairs and more space between the yarns might enhance the air permeability of the fabrics. In the impregnation method, the pores in the fabric are covered with the applied nanoparticle and thus the gaps are closed. But, in alternative application method, the pores are not closed by rGO nanoparticles and hence, it is possible to obtain higher permeability values.

Table 8
ANOVA LSD test results of air permeability values of the fabrics knitted from undoped and rGO doped yarns

Yarn types		Sig.
Undoped	rGO doped-65 mL/h	0.000*
	rGO doped-70 mL/h	0.000*
	rGO doped-75 mL/h	0.000*
rGO doped-65 mL/h	rGO doped-70 mL/h	0.103
	rGO doped-75 mL/h	0.040*
rGO doped-70 mL/h	rGO doped-75 mL/h	0.606
*. The mean difference is significant at the 0.05 level.		

In fabric finishing treatments such as impregnation, coating, vapour deposition etc. methods, air permeability of the applied fabrics generally decreases due to coating of the fabric and presence of nanoparticle in the fabric structure. Nanoparticles block the passage of air and the pores in the fabric structure are closed. This results in a decrease in air permeability and hence the properties related with fabric comfort. It is desirable to have a fabric with higher air passage to facilitate heat dissipation and sweat evaporation (Chen et al. 2010). Summing up, alternative application method provided an important advantage in air permeability and hence the properties related with fabric comfort.

Overview Of Ecological Impact

Textile industry has important environmental impacts such as high energy consumption, water and air pollution, waste disposal and odour generation (Butekom 2014). Among the textile processes, in particular, dyeing and finishing processes cause high amount of natural resource consumption and wastewater discharge comprising high amounts of chemical substances. Especially, in recent years, dyeing-finishing processes have become even more important with the increasing expectations of consumers from textile products. This results in the production of wastewater arising not only from the removal of impurities from the raw materials but also from the residual chemical reagents used in processes. Therefore, large amounts of water are generally required for processing (Correia 1994). For example, water consumption reaches to 80–100 m^3/ton of finished textile and waste water discharge is about 115–175 kg of COD/ton for a finished textile product. Therefore, biggest impact of finishing processes on the environment is related to primary water consumption and waste water discharge (a large range of organic chemicals, low biodegradability, colour, salinity) (Savin et al. 2008).

Nowadays, the shortage of water resources is severe in the world and clean water has become increasingly scarce as discharges of industrial effluents. Textile wastewater includes a large variety of dyes and chemicals additions (detergents, sulphide compounds, solvents, heavy metals, inorganic salts etc.) and this case makes the textile industry an environmental threat in terms of liquid waste and also its

chemical composition (Venceslau et al. 1994). Therefore, negative effects of the textile industry on human health and natural life have reached a nonignorable level. From this point on, in the textile industry, there is a need to use production methods and materials that have lower environmental impacts in terms of chemical, energy and water consumption and waste water generation.

In the study, ecological impact of the developed alternative application method was evaluated in general with the finishing methods widely used in practice. Finishing processes used in practice involve impregnation of the fabric using a padding technique followed by a fixation step by heat. Subsequent washing may be carried out to remove residual chemicals. However, in developed application method, drying/fixation and subsequent washing processes are not required. On account of no need of drying/fixation and additional washing, the method actually provides an advantage. When the water consumption was taken into consideration, water consumption varies between 0.5-10 litres per 1 kg of fabric depending on fabric and machine types in impregnation and exhaustion methods widely used in textile dyeing and finishing operations (Tarakçıoğlu 1979). Güngör et al. (2009) reported that 100 litres of water is averagely consumed per 1 kg of textile product in textile dyeing and finishing processes. Lu et al. (2010) stated that about 150 litres of water are consumed on the average for every kg of cloths processed in a typical dyeing and finishing mill. However, in alternative application method, it is between 0.4-0.5 litres per 1 kg. In addition, in other application methods such as impregnation, exhaustion and coating, substances that cannot be taken by the fabric are thrown away, and hence negative effects such as environmental load caused by wastewater and inefficient use of functional materials arise. In the alternative method, unused solution was not disposed since the sample was not kept in a solution or passed through a solution as in conventional application methods such as impregnation, exhaustion etc. Therefore, the alternative application method may offer significant advantages, especially in terms of clean water usage and wastewater disposal, compared to commonly used application methods such as impregnation, exhaustion and coating.

Table 9
Ecological evaluation of application methods used in textile dyeing & finishing processes

Method	Application conditions		Ecological situation
Impregnation	Short flotte (1/0.5 - 1/1)	-Water consumption (min. 0.5 L for 1 kg fabric)	-Dispersion, which is not absorbed by the fabric, turns into waste water together with the chemicals.
Exhaustion	Long flotte (1/3 - 1/50) Generally, 1/8 or 1/10 flotte rate is used.	-Water consumption (min. 3-10 L for 1 kg fabric)	-The fabric cannot incorporate the functional material in the bathroom. -Efficiency is low. -After application, a significant amount of wastewater occurs.
Alternative method	-	-Dispersion is applied onto the fiber strand in the yarn spinning zone. -The amount of water used for 1 kg of fabric is approximately 0.4-0.5 L.	-Water consumption is lower than current methods. -There is no apparent wastewater problem.

4. Conclusions

In present study, it was worked on an alternative method to make the fibre-based structure conductive for the production of conductive textile products. Reduced graphene oxide nanosuspension (rGO) synthesized with single-step process was fed on to the viscose open fibre bundle from 30 mL/h to 80 mL/h by a developed feeding apparatus and 70 mL/h was determined as an optimum feeding rate due to the lowest resistance value, better resistance stability values besides comparable tensile properties with undoped reference viscose ring spun yarns. Additionally, SEM images of rGO doped viscose ring spun yarns indicated the deposition of a certain rGO flakes on the fibre surface. On the other hand, experiments on viscose and also cotton and polyester fibres showed that alternative method provides to impart certain degree yarn conductivity to the different fibre types. After the winding and knitting processes, as expected, electrical resistance values increased due to yarn rubbing with various parts of winding and knitting machines. However, the yarns on bobbin form and knitted fabrics had still electrical conductivity feature even after high friction. Additionally, slightly change in resistance values to initial resistance value ($\Delta R/R_0 \approx 1$) indicates that electrical resistance values of yarn, bobbin or knitted fabric forms are almost similar. In addition to effect of mechanical effects on yarn conductivity, it was also determined that rGO-containing fabrics kept their conductivity property after laundering. The fabrics had considerably low resistance values than 100 kΩ before and even after washing. Due to the protection of fabric porosity, the fabrics produced from alternative application method had higher air permeability values and hence more permeable structure than the fabrics knitted from undoped yarns. Unlike the impregnation method, the pores in the fabric were not closed by rGO nanoparticles and hence, this case might allow to obtain breathable electronic textile structures. On the other hand, rGO included fabrics obtained with alternative application method have comparable electrical resistance values with immersion&drying method (5

repetitions). In addition to performance properties of alternative application method, there was no need of drying/fixation, washing processes and discharging of unused solution. Therefore, it is believed that alternative application method may offer significant advantages in lower clean water usage, wastewater disposal and environmental load.

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Tables

Table 7 is not available with this version

Figures

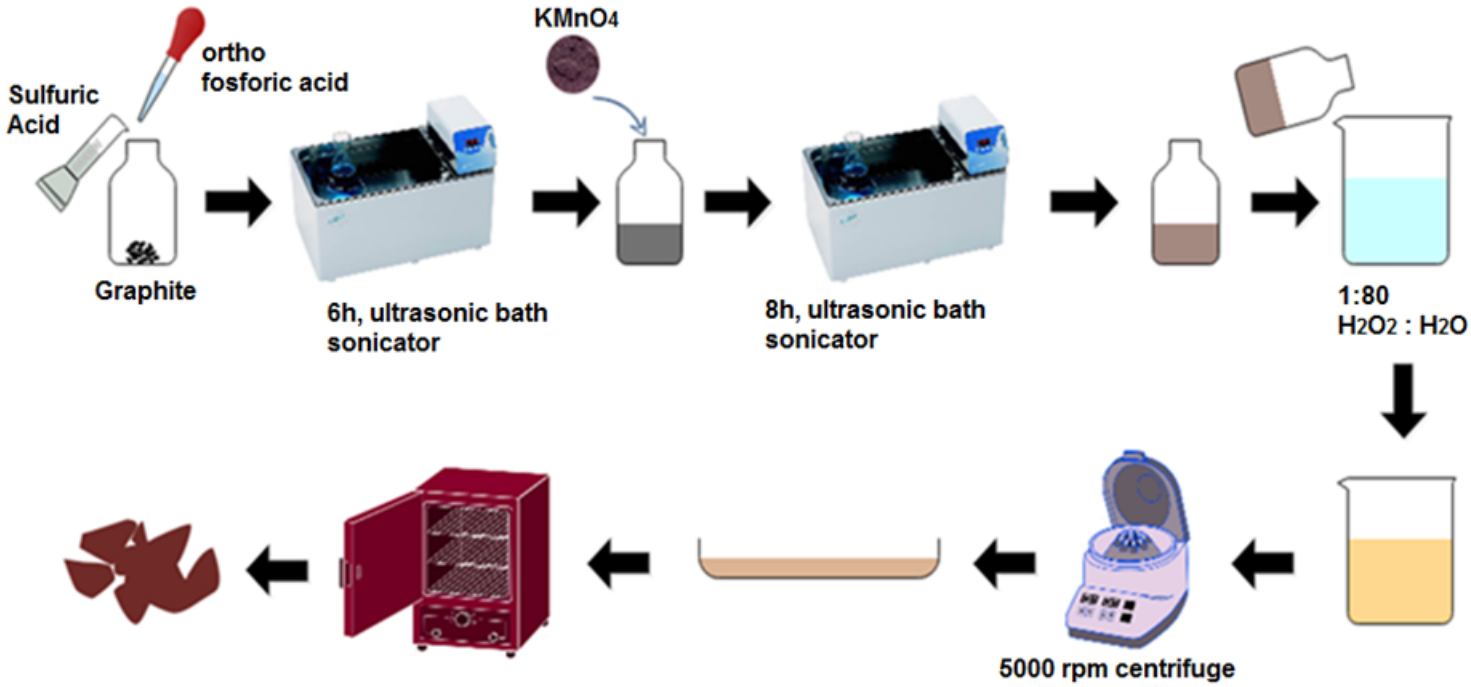

Figure 1

Schematic representation of graphene oxide synthesis

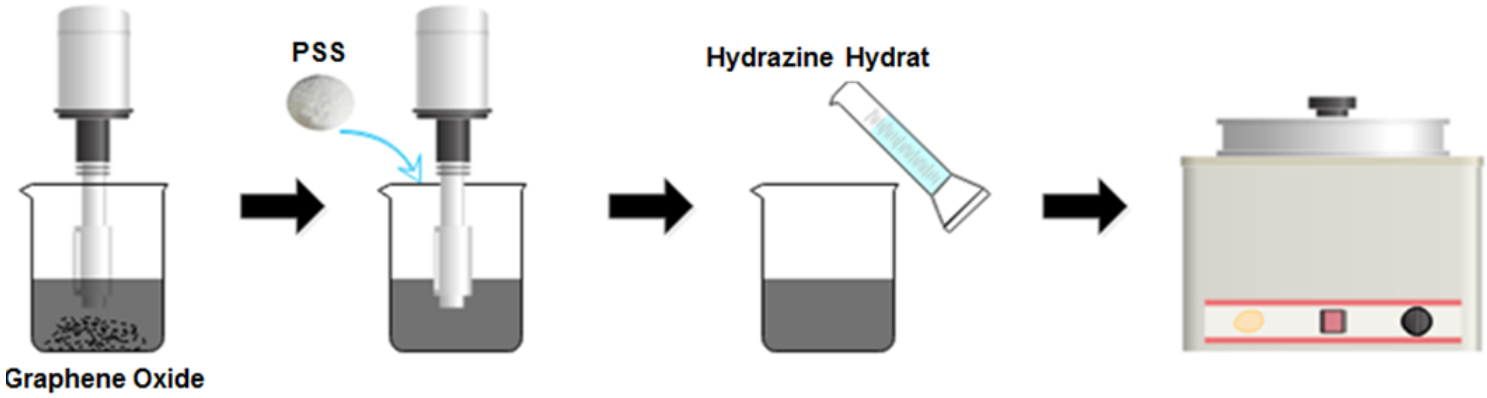

Figure 2

Schematic representation of the reduced graphene/poly (sodium 4-stylenesulfonate) suspension production

Figure 3

Production of rGO-containing conductive yarn in the conventional ring spinning system

Figure 4

SEM images of the undoped reference (a) and rGO doped (b) Ne 16/1 viscose ring spun yarn.

Figure 5

Reference (a) and rGO applied (b) viscose ring spun yarn appearances

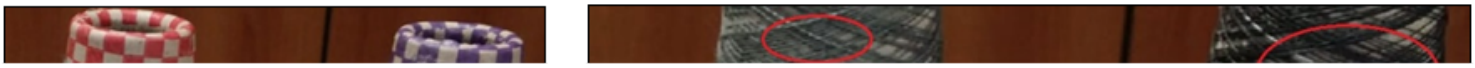

Figure 6

Irregular colour distribution resulted from insufficient rGO nanosuspension feeding

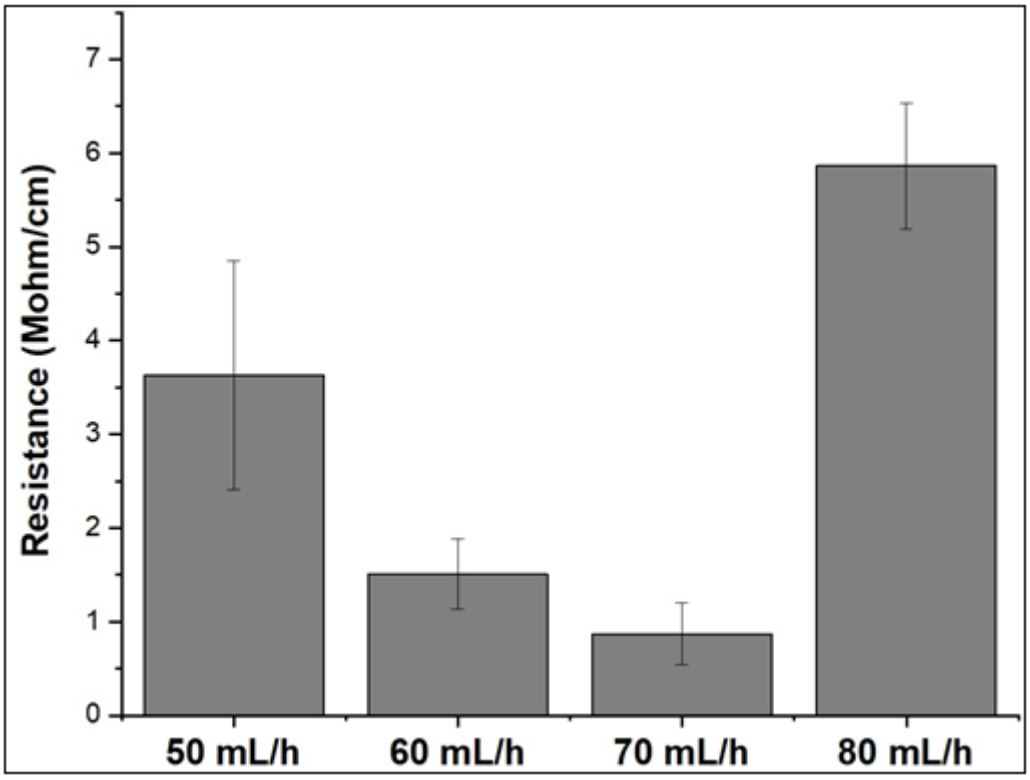

Figure 7

Electrical resistance results of rGO/viscose ring spun yarns for different rGO nanosuspension feeding rate values

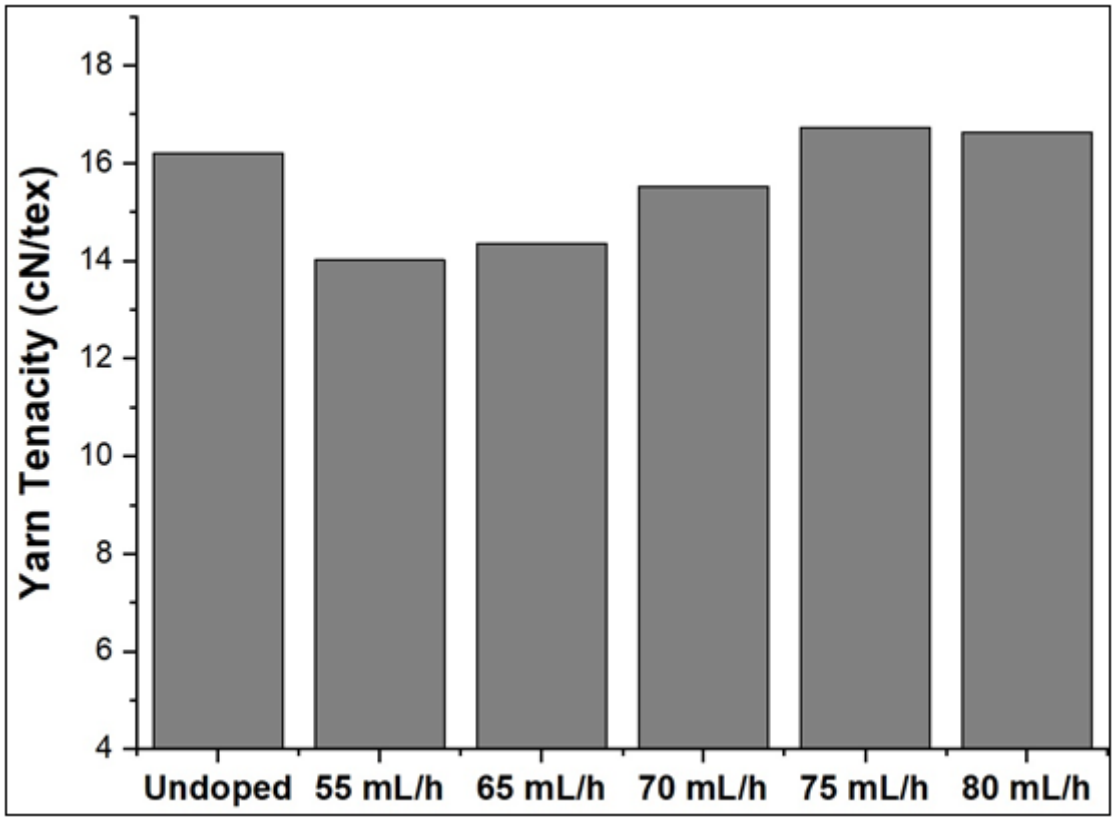

Figure 8

Tenacity results of undoped and rGO nanosuspension doped viscose ring spun yarns

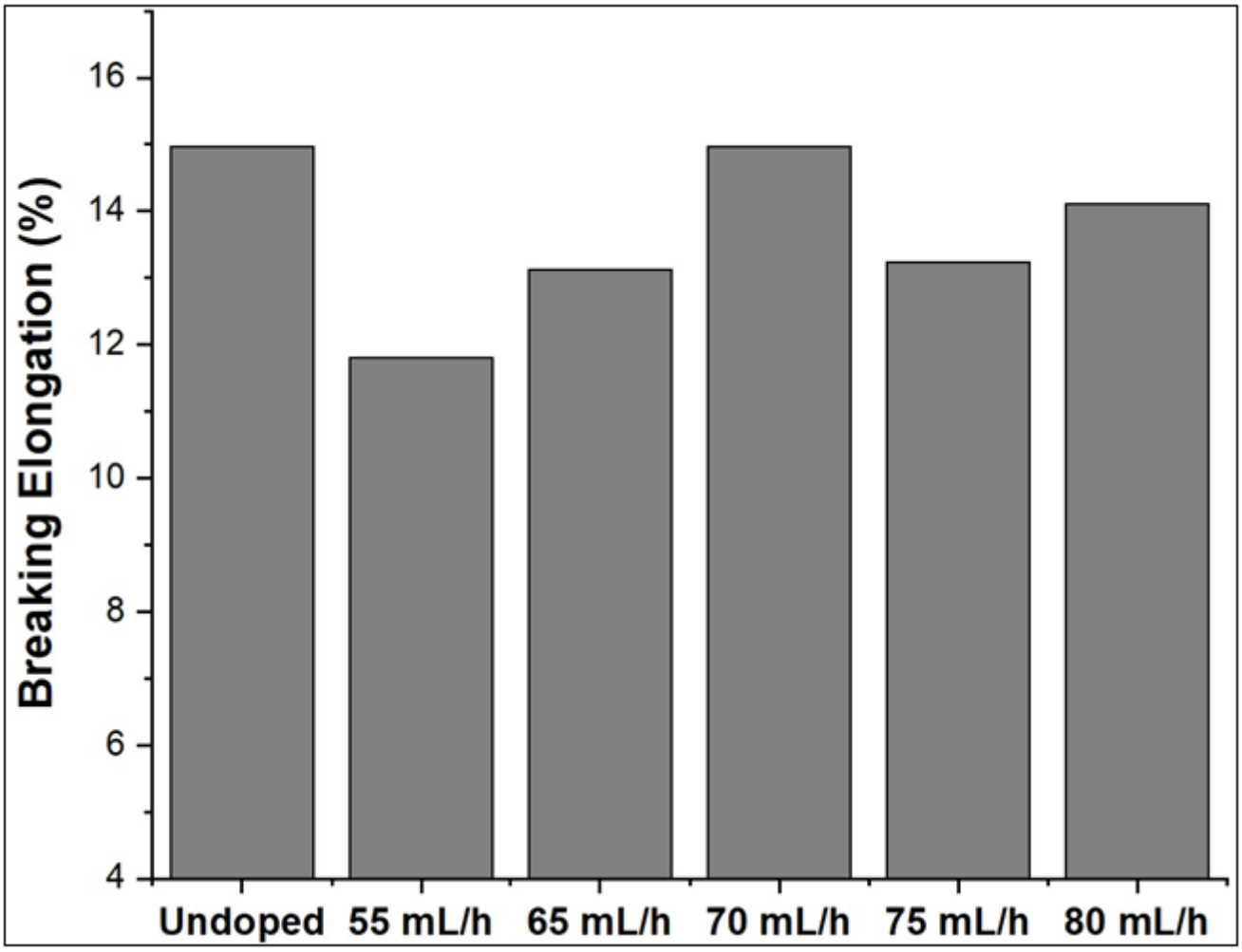

Figure 9

Breaking elongation results of untreated and rGO nanosuspension doped viscose ring spun yarns

Figure 10

rGO doped yarn bobbins after winding process

Figure 11

Fabric samples knitted from undoped and rGO doped viscose ring spun yarns

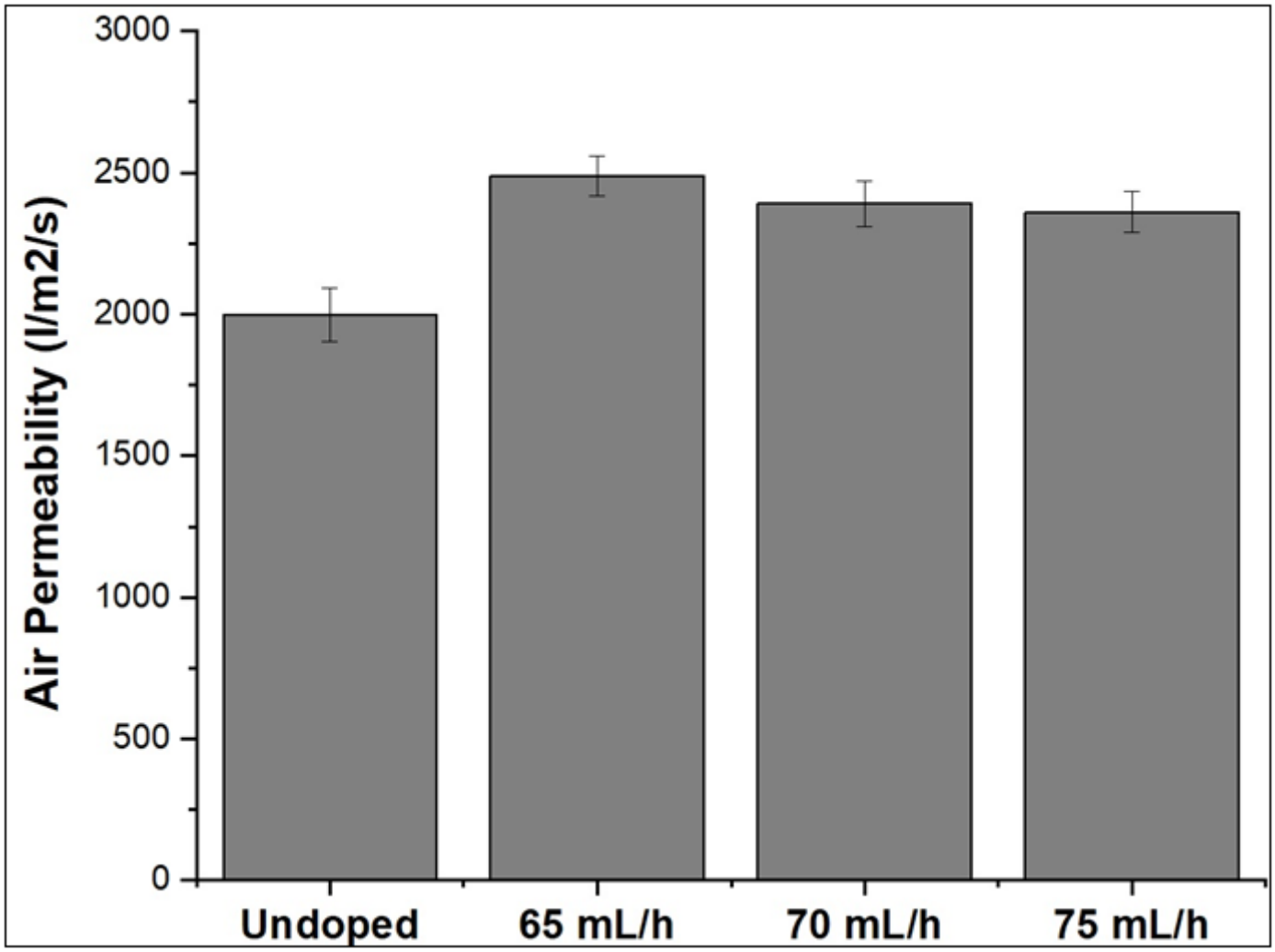

Figure 12

Air permeability results of knitted fabrics obtained from undoped and rGO doped yarns

Figure 13

Reference (a) and rGO doped (b) viscose ring spun yarns