Mechanical characterization of functional graphene nanoplatelets coated natural and synthetic fiber yarns using polymeric binders

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ABSTRACT
Fabrication of electrically conductive yarns (glass, flax and polypropylene fibers) coated with graphene nanoparticles (GNP) were characterized for their mechanical properties and compared with their electrical properties. The composites were produced with the use of polymeric binders (epoxy resin and thermoplastic starch) and two different dip-coating methodologies were developed to create the coating layers. Technique-1 involved coating of binder and then GNP layer whereas Technique-2 had a mixture of binder and GNP in the predetermined ratio, which was coated on the yarns. The mechanism of adhesion varies or influences on a number of factors such as the nature of the fiber surface, coating method and effective binder. Tensile properties of the yarns were measured by an appropriate standard, and the highest tensile strength was noticed with epoxy-based glass fiber samples as 222 MPa followed by flax fiber samples as 206 MPa. The composites of starch-based showed poor mechanical performance compared to those of epoxy ones. This was due to poor adhesion between the surface and starch layer (interphase) where the Van der Wall’s force was quite low. Electrical conductivity, glass fiber yarns with epoxy binder were identified to have the highest electrical conductivity of 0.1 S.cm$^{-1}$ among other samples.

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1. Introduction

Wearable electronics utilizing fiber yarns and fabrics have become a great interest in the materials science community owing to their useful applications in cheap electronic devices such as wearable displays, motion sensors and flexible conducting wires [1–4]. Carbon-based materials, especially graphene and its derivatives have garnered good attention and found to be good candidates to fabricate such devices. Natural and synthetic fibers yarns have been considered by various industries these days due to their potential properties abundant availability, which can potentially be employed in electronic devices [5]. However, turning fiber yarns into conductive materials using graphene or similar nanoparticles is always critical due to the complexity in coating methodologies [6–8]. Due to these reasons, there have only been a few studies reported and there is a significant gap to understand their fabrication processes, properties and potentials. Additionally, there is no much attention to the use of natural fibers in such application. However, there are several studies on developing conductive coating using nanomaterials such as graphene [9], carbon nanotubes (CNT) [10,11], nickel, indium dioxide [12] and carbon black [13], but their coating methods were expensive and complex.

Conductive glass fiber yarns were produced by Zhang et al. [2] with use of CNT coating through electrophoretic deposition (EPD) in which a homogeneous coating of CNT was done on the glass fiber, but the fabrication process involves multiple steps before reaching the final state EPD and the conductivity was only reported to be ~ 1 × 10⁻⁴ S.cm⁻¹. Tzounis [14] reported that glass and jute fibers modified, not a coating, with MWCNT through synthesizingCNT based colloidal system and depositing on the fiber surface. Yet again, the process heavily involved synthetic chemical deposition that involves wet chemicals while electrical conductivity was achieved only as 6.67 × 10⁻⁵ S.cm⁻¹ with single glass fiber yarns. The jute fiber yarns had even much lower electrical conductivity. In a study by Rausch et al. [15], glass fibers were immersed in CNT dispersion (in the controlled deposition of CNT particles) to create a conductive layer resulted in improved mechanical properties but exhibited poor electrical conductivity only up to 1 × 1⁻¹⁰.

Generally, for creating a coating layer on the fiber surface, it is essential to strengthening the interphase where the hierarchical structure builds up strongly and an electron transporting channels are effectively created. Upon creating such strong interphase (where the Van der Wall’s force higher) a large surface area of active materials, these conductive particles, would retain their original properties [5,16–18]. Several studies [19,20] have showcased that surface modification of fibers, particularly for natural fibers, the adhesion between fiber-matrix interphase is significantly improved. A number of chemical modifiers are available for natural fibers and in particular, silane-based surfactants are highly compatible with natural fibers enhancing its adhesion with the polymer matrix [21]. Hence, the fibers used in the study were treated with silane surfactant.

Carbon allotropes are potential conductive fillers that can be used to develop conductive composites [22,23]. Amongst such materials, graphene and carbon nanotubes are proved highly conductive. However, we chose graphene due to health reasons, as CNT are quite carcinogenic [24,25].

Herein, we report simple dip-coating techniques to develop electrically conductive wires using GNP, different fiber yarns (flax fiber, glass fiber and polypropylene fiber) as wires and epoxy resin and thermoplastic starch as binding materials. Two variations of
dip-coating approaches were utilized to see the effect of the fabrication process, and the electrical conductivity, mechanical properties and morphological studies were carried out and results were compared. When performing a systematic approach by choosing the right material combination/proportion and manufacturing methods with a focus of optimizing coating process parameters, it is possible to enhance both mechanical and electrical properties together.

In all the previous studies mentioned above, it was indicated that the interphase between the fiber surface and nanoparticulate coating was poorer, which is the primary reason why electrical conductivity did not meet the practical outcome. This is directed us to use the polymeric binder through which the adhesion between the fiber surface and the nanoparticles was expected to be improved drastically. Moreover, none of these studies attempted to produce continuous fibers yarns coated by the conductive layer of nanomaterials or conductive materials. Furthermore, a comparative study of both mechanical and electrical properties by varying different factors (such as different fiber yarns, binders and coating methods) were considered in this study whereas other reported studies did not have. This comparative analysis would give valuable insight to identify potential areas of application. By using simple and cheap materials with very low loading of GNP, we are providing evidence that inexpensive natural fibers can be easily fabricated. Furthermore, the knowledge of effects of binders (thermoset and thermoplastic) on the electrical and mechanical properties of the fiber yarns have been extended/investigated by using epoxy as thermoset binder and thermoplastic starch (TPS) as a thermoplastic binder.

2. Experimental

2.1. Materials

Flax fiber yarns and glass fiber yarns (91,106, Wrap – EC 5–11 tex x 2 and Fill – EC 5–11 tex x 2) were purchased from Jayashree Textiles, India, and Interglas, respectively. Polypropylene yarns from H5300 granules manufactured by Honam, South Korea, were produced in-house. Epoxy resin (Prime 20LV) and slow hardener (Prime 20) were bought from Gurit, New Zealand. Thermoplastic starch (85,652) and graphene nanoplatelets (GNP) were purchased from Sigma-Aldrich, New Zealand and Emfutur, Spain, respectively.

2.2. Fabrication of composite conductive fiber yarns

The two types of dip-coating processes are depicted in Figure 1. Technique-1 was a layer by layer coating approach where the binder (epoxy/thermoplastic starch) was coated on the fiber surface followed by a layer of GNP. The coated samples were then cured in a vacuum oven at 60 °C for 3 hours. Technique-2 involves the coating of the predetermined mixture of GNP and the binder on the fiber yarns. The proportion of GNP in the binder was determined by the simple rule of mixtures as showed in the graph in Figure 2. Prior to the coating process, 1:1 molar ratio (3-Aminopropyl)triethoxysilane (APTES) in ethanol was used as a surfactant to treat all the fiber yarns (while no improvement in PP yarns was expected) to enhance adhesion at the interphase potentially. The fibers were immersed in APTES aqueous solution at 30 °C for 2 hours. No further pH adjustment was done with the silane solution.
The ratio of epoxy resin to slow hardener was 100:26 whereas the thermoplastic starch being a high molecular mass carbohydrate- \((C_6H_{10}O_5)_n\) the mixed in distilled water as a percentage solution rather than as a molar strength solution. For both coating techniques, the pull rate was considered critical and a separate experiments were made in order to determine the most practical pull rate by varying the same as 50, 75, 100, 150 and 200 mm.min\(^{-1}\). The conductivity was then checked at these different pull rate where the electrical conductivity was generally improved or incorporated at a slow pace. The conductivity started dropping from its highest value achieved after 150 mm.min\(^{-1}\) and further drop at 200 mm.min\(^{-1}\). Therefore, we fixed the pulling rate at 100 mm.min\(^{-1}\) as the desired rate of pulling the fiber yarns through the coating materials. The thickness of the epoxy-based and starch-based coating layers are 0.5 mm and 0.8 mm, respectively.

2.3. Characterization

**Tensile testing** was performed in an Instron-5567 according to ASTM D3822. From the availability of different load cells (2, 5, 20 and 30 kN), accurate measurements were ensured. The Instron was equipped with a high-resolution digital camera to amount strain by tracing contrasting gauge marks positioned on the specimen. Blue-Hill materials testing software was used to control the machine and testing. The crosshead speed of the tests was 5 mm.min\(^{-1}\) according to the standard.

**Electrical conductivity** values of the samples were measured using a 4-point conductivity tester in which the probes were made of brass coated with gold, and the distance between each probe was 18 mm.

Both mechanical and electrical test results were averaged from 5 replicates for each sample.

**Scanning electron microscopy (SEM)** images were captured from an FEI Quanta 200 FEG, USA, armed with an EDS Detector of Si-Li (Lithium-drifted) with a Super Ultra-Thin
The samples were sputter-coated with a Quorum Q150RS sputter coater and the normal target used was Pt or Au.

**Thermogravimetric analysis (TGA)** analysis was performed using a TGA Q5000 IR instrument at the heating rate of 10°C.min⁻¹ (Ballistic heating > 1000°C.min⁻¹). The samples were characterized in the temperature range of 0 to 800°C.

Figure 2. Stress-strain curves of neat fiber yarns and the composite yarns.
3. Results and discussion

3.1. Tensile properties

Typical stress-strain curves of different samples can be seen in Figure 2. It can be observed that almost all the composite samples behaved in a very similar way to that of its neat materials with the increment in strength with the layer of coating. In the overall observation, each addition of coating layers (binders and GNP in Technique-1 and the blend of binder and GNP in Technique-2) improved the mechanical strength along with the electrical conductivity of the yarns. The composite yarns display almost a linear behavior in the elastic regions and then they meet a swift drop at their breakage points after reaching their maximum tensile strength. Starch-based samples (for flax, glass, and PP fibers) exhibited the low stiffness compared to those of epoxy-based which is due to the improved adherence of epoxy and fiber at their interphase [19,20]. Starch-based samples also showed poor electrical conductivity values which are obviously because of breakage in the conductive network where GNP have no interparticle connection.

Similar behavior was observed for the ultimate tensile strength of the coated samples. UTS of ~225 MPa was the highest strength with glass fiber-epoxy combination produced by Technique-1 whereas the lowest strength was exhibited by flax fiber-starch combination as 116 MPa, Figure 3. This further confirms that lower ultimate tensile strength that could be due to the weaker bonding between the GNP particles [26] and starch on the fiber yarns. However, the percentage increase of UTS in all kind of fiber yarns samples is highest with starch-based than those of epoxy-based. This could be due to a high peak to valley points of starch coating layer whereas epoxy coating layer is comparatively thinner. The summary of the mechanical properties of different samples is presented in Figure 3. These results suggest that the degree of adhesion between fiber yarns and the polymer coating layer could substantially affect the mechanical properties of the conductive yarns.

Figure 4 (a) shows the electrical conductivity values of the samples and their standard deviation and Figure 4 (b) compare different samples with their UTS and electrical properties. Both two techniques have certainly incorporated conductive network on the fiber yarns, but comparatively, Technique-1 has produced better results through optimal coating layer. The conductivity measurements also showed they the values stayed constituents form the 5 replicates with a standard deviation of ± 4% and ± 7% for epoxy-based and starch-based, respectively. As mentioned earlier, both dip-coating techniques were effective in establishing the conductivity on non-conducting fiber yarns, but it highly depends on the choice of binding material.

Mechanical interlocking [27,28] and interfacial adhesion [29,30] played a vital role in the variation in electrical conductivity as strongly as they play their roles in improving the mechanical properties of the yarns. Particularly, it could be possible that the interphase between fiber yarns and binders was strengthened by the coupling agent (silane). It is known that an extent of silane molecules reacts with the hydroxyl functional entities on the surface of the fiber yarn creating polysiloxane groups or a layer of the polysiloxane. This layer provided a strong covalent bonding for the samples having a higher electrical conductivity (and also mechanical properties) [31].

From these observations, it can be said that electrical conductivity and mechanical properties of glass fiber/flax fiber/PP yarns are directly proportional to the degree of
adhesion and interlocking effectiveness which are in turn depending on the nature of binding material. For epoxy-based samples, both Technique-1 and Technique-2 resulted in similar electrical conductivity. The pre-suspension of GNP in the binder (Technique-2), the weight percentage of GNP can be adjusted, although only one wt.% case was used to demonstrate the method’s viability.

3.1.1 Effect and mechanism of addition of binder and GNP
The effect of polymer binders played a vital role in the established electrical conductivity of the fiber yarns while simultaneously improving their mechanical properties. By closely
Figure 4. (a) Electrical conductivity and standard deviations GNP-coated samples produced by Technique-1 and Technique-2 and (b) comparative plot of UTS and electrical conductivity of the samples.
coating the GNP particles is highly dependent on the chemical compatibility of the binder and the amount of binder [32] on the surface of the fiber, which eventually influences the properties of the composite material. Between the two of these factors amount of fibers was assumed to be controlled by the limiting hole through which the fiber yarns were pulled out to create the binding and conductive layers. The chemical compatibility is certainly of a higher degree in epoxy-based samples than those of starch-based ones. The starch-based samples adhere loosely on the surface of the fiber yarns and also binds the GNP particles similarly, due to poor adhesion and a high number of dynamic bonds of starch (since starch layer gets damaged or ripped off after curing) compared to strong static bonds in epoxy resin after curing. This could be attributed to a lower degree of mechanical interlocking [32] between the fibers and starch layer.

On the contrary, it is clear that the mechanical interlocking is significantly higher at the epoxy and fiber interphase, which was evident from their higher mechanical performance and electrical conductivity. The comparative plot shows that there is a difference in trends between electrical conductivity and UTS. This is mainly attributed to breakages in the conductive layer which do not have a significant effect on the mechanical strength but electrical conductivity.

3.2. Microstructural analysis

Surface morphologies of glass fiber/flax fiber/PP yarns coated with binders and GNP are shown in Figure 5. The obvious difference between the epoxy-based and starch-based samples is that the significant irregularity of starch-based samples which is closely ascribed to their lower electrical and mechanical properties. These irregular surfaces indicate an irregular GNP conductive layer is influencing large-scale charge transfer negatively. A comparable surface microstructure was noticed by Liu et al. [33] with their samples made of cotton yarns with Ni and rGO by an electrochemical coating method.

These breakages, as depicted in schematics (Figure 6), on the surface of starch-based samples, also indicate that they possess poor interfacial adhesion/strength than those of epoxy-based ones. As epoxy-based samples have significant epoxide groups, which tend to chemically react with the carboxyl-functional groups present in GNP, which would result in better the interfacial strength. As indicated previously, the silane coupling agent also reacts with epoxide moieties, epoxy-amine, and silanol functionalities giving more stability to GNP and epoxy coating layers [34,35].

3.3. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed to quantify the mass consistency ratio of the different coating layers on the fiber yarns by the two different binders and two different coating techniques. Figure 7 shows the thermograms of the different samples from a temperature range of 0°C to 800°C. To analyze the graphene weight percentage in Technique-1, the difference between the samples with and without GNP layers was compared, and it can be seen that the difference in the graphene loading was greatly controlled during the fabrication process as there was only a negligible difference noticed. Similarly, the mass of binders (epoxy and starch) was assessed to be consistent as depicted by the respective thermograms. For Technique-2, the difference was
determined in a similar approach where thermograms of neat fiber yarns and fiber yarns coated with the conductive layer (a mixture of binder and graphene) were compared and the weight loss difference was again noticed to be very consistent reflecting the mass loading was similar in different samples. This analysis also indicates that the weight loss between samples without ‘conductive layer’ and with ‘conductive layer’ (for epoxy and starch-based composites) was 15% within the temperature boundary. For Technique-2, the average weight loss of 23-24% between neat fiber yarns and the layer of ‘binder and GNP’ was recorded.

4. Conclusions

Mechanical properties of conducting fibers yarn from flax fiber, glass fiber, and PP produced by two different cheap and efficient coating processes were assessed. Thermoset (epoxy resin) and thermoplastic (thermoplastic starch) binders were used to bind GNP on the fiber surface. Factors responsible for influencing mechanical properties and electrical properties were analyzed and most critical factors were found to be interfacial adhesion, uniform coating layer and type of fabrication process. The ultimate tensile strength was higher in epoxy-based samples than those of starch-based ones due to starch being a poor binder, which has significant irregularity and breakages in the coated layer, which was observed in SEM analysis. The Technique-1 fabrication process
was better compared to Technique-2 in terms of achieving uniform GNP coating which contributed to improved mechanical and electrical properties. For Technique-2, a pre-determined GNP weight fraction of 6 wt.% was used as an optimal mass to reach the highest possible electrical conductivity. Among all three fiber yarns (GF, PP, and flax), glass fiber and the epoxy combination resulted in highest UTS as \(-225\) MPa and lowest was observed in starch and flax combination. The electrical conductivity values were however had a different scenario with only slight differences in them (GF-0.1, PP-0.08, and FF-0.06 S.cm\(^{-1}\)). Starch was primarily considered to advantage environmental aspects of these materials, but the results observed for starch-based samples did not meet the expected standards owing to poor compatibility.
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Disclosure statement

No potential conflict of interest was reported by the authors.

Figure 7. Thermograms of different composites from Techniques 1 and 2 (*GF = glass fiber, FF = flax fiber and PP = polypropylene yarn).
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Novelty Statement

The work is based on the fabrication of graphene nanoplatelets fibre yarns through efficient coating methodologies. The purpose of the research was to develop cheap conducting wire by incorporating electrical properties to natural (waste renewal as well as value added) fibre yarns. Both synthetic and biodegradable binder was utilised to deposit nanographene on to the surface of fibre yarns. A considerable electrical conductivity was noticed in different samples produced. The attempt to produced biodegradable conducting yarns with starch-based binder has relatively lower conductivity than those of other samples based on epoxy binders.

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