



# Graphene nanoplatelets loaded polyurethane and phenolic resin fibres by combination of pressure and gyration



A. Amir<sup>a</sup>, S. Mahalingam<sup>a</sup>, X. Wu<sup>a,b</sup>, H. Porwal<sup>c</sup>, P. Colombo<sup>d</sup>, M.J. Reece<sup>c</sup>, M. Edirisinghe<sup>a,\*</sup>

<sup>a</sup> Department of Mechanical Engineering, University College London, London WC1E 7JE, UK

<sup>b</sup> School of Materials Science and Technology, China University of Geosciences, No.29 Xueyuan Road, Beijing 100083, China

<sup>c</sup> School of Engineering and Materials Science, Queen Mary University of London, Mile End Road, London E1 4NS, UK

<sup>d</sup> Dipartimento di Ingegneria Industriale, Università di Padova, Via Marzolo, 9, 35131 Padova, Italy

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## ABSTRACT

A simple and effective process combining pressure and gyration has been developed and used to jet-out composite fibres containing graphene nanoplatelets. Thermoplastic polyurethane and phenolic resin polymers were used as matrices. Processing parameters such as rotation speed, pressure and polymer concentration had a marked influence on the composite fibre diameter. Focussed ion beam milling and etching verified the effective incorporation of the graphene nanoplatelets into the composite fibres. Morphological, rheological, physico-chemical and thermal properties of the composite fibres were evaluated to identify possible applications for them.

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## 1. Introduction

Hybrid polymer composites have attracted significant interest in the field of materials science and engineering over the years. Composite material may have new or enhanced properties compared with their pristine individual components. For example, incorporation of carbon fibres into a thermoplastic polyurethane polymer matrix can develop a composite structure with high Young's modulus, better strength, good thermal conductivity and excellent electrical properties [1]. Carbon-phenolic resin polymer composite based bipolar plates were shown to have high electrical conductivity and good mechanical properties and are used in proton exchange membrane fuel cells, which are an attractive power source for portable, stationary and transport applications [2]. Graphite reinforced thermoplastic polyurethane composite was reported to have excellent shielding effectiveness, which make

them invaluable as shielding materials in commercial applications such as enclosures of electronic devices, electronic packaging and military devices [3]. A dramatic enhancement of fracture properties (fracture toughness and energy release rate) was obtained in graphene-EPON 862 thermoset polymer composites in comparison with baseline polymer [4].

Graphene is a single atom thick sheet of graphite that is self-assembled through Van der Waals forces and consists of  $sp^2$  carbon lattices regarded as 2D material with superior properties and negligible thickness [5]. Two dimensional graphene (G) and graphene based materials have attracted tremendous interest in the past decade owing to their superior surface area and show exceptional physical properties such as mechanical strength, good electrical and electrochemical activity [6–9]. Graphene is an excellent choice for a super-capacitor electrode material enabling high power density, high energy density and stable cyclic performance [10–12]. It has shown great promise in environmental remediation [13]. In addition, graphene can be an antibacterial material in bioengineering to prevent attachment and proliferation of microbes without the concerns of environmental pollution [14].

\* Corresponding author.

E-mail address: [m.edirisinghe@ucl.ac.uk](mailto:m.edirisinghe@ucl.ac.uk) (M. Edirisinghe).

Polyurethane is a thermoplastic polymer which has received increased interest nowadays due to manufacturing versatility and improved strength and stiffness [15]. The recyclability of these materials is an added advantage, and with other features such as biocompatibility and biodegradability it is very useful and suitable for tissue engineering of cardiovascular systems [16]. Phenolic resins are widely used in building various functional materials and coatings due to their high mechanical strength, electrical insulation, flame retardant resistance and low thermal conductivity [17]. Combining these polymers with graphene has generated a great deal of interest as they show improved mechanical and thermal properties [18].

Manufacturing of graphene based polymer composites involves finding a suitable blending method to reach a satisfactory dispersion of graphene throughout the polymer matrix. Generally, there are three methods to process graphene-based polymer composites, namely in situ polymerisation, solution compounding and melt blending [19]. In-situ polymerisation comprises polymerisation of a monomer and/or an oligomer in the presence of filler graphene and combined with in-situ compounding technology confers stronger interactions between the reinforcing graphene and the polymeric phase [20]. This method exhibits better mechanical properties and a low percolation threshold. However, it needs high electrical energy to disperse the graphene in the polymer matrix and may not be suitable for mass production of composites. In solution compounding, the polymer is dissolved in a solvent and filler is dispersed in the resulting solution. After mixing, the solvent is removed and the bulk polymer containing the graphene is used to give shape to the composite [21]. It also has an advantage of lower percolation threshold than the in-situ polymerisation method, however, the use of large amounts of solvent and the associated environmental pollution has prevented this method being used to mass produce polymer composites. Melt blending involves melting of the matrix polymer and then graphene is mixed in with traditional mixing equipment such as an extruder, internal mixer or two-roll mill. It is a preferred industrial technique as it is a direct, cost effective, and environmentally friendly process since no solvent is involved [22]. However, the percolation threshold values attained using this technique are higher than for the in-situ polymerisation and solution compounding methods.

Recently, pressurised gyration has shown great promise in forming nano- and micro-assemblies such as nanofibres, composite nanofibres and microbubbles and capsules [23–27]. This technique offers consistency, reliability and is easy-to scale up [23–25]. In typical pressurised gyration of a polymer solution, the centrifugal force and the fluid flow overcomes the withholding surface tension force to initiate instability in a liquid jet that subsequently breaks up into fibres. Finally, the evaporation of the solvent leads to solidification of the fibres formed [23]. The fibre size distribution and morphology are influenced by process parameters such as rotating speed, working pressure and the physical properties of polymer solution [23]. In addition, studies of polymer-protein solutions have shown that pressurised gyration is capable of generating microbubbles [25]. In a typical parametric plot of rotating speed versus pressure, it has been found that a minimum rotating speed and pressure are required to form a structure. Below this critical minimum rotating speed and pressure structures are not formed due to insufficient forces to initiate an instability [25]. Moreover, there is also a window of rotating speed and pressure required to form fibres and in other instances, microbubbles.

In this work, we used thermoplastic polyurethane and phenolic resin in combination with sonicated graphene nanoplatelets to form hybrid polymer composites by pressurised gyration. The aim of the present work is to develop a novel processing route to directly generate well dispersed polymer-graphene composite

fibres which could be used in applications such as bipolar plates in proton exchange membrane fuel cells and as shielding materials in electronic packaging. Therefore, the morphological and structural features and properties of the composite fibres were extensively studied.

## 2. Materials and method

### 2.1. Materials

Thermoplastic polyurethane (TPU, Desmopan DP 9855DU) and phenolic resin (PR) were supplied by Bayer Italia (Milan, Italy) while polyvinylpyrrolidone (PVP) supplied by Sigma Aldrich, UK and were used as the polymer matrices. Graphene nanoplatelets were supplied by XG Sciences (Lansing, Michigan, USA) and were used as a filler material with a typical size of 2–3  $\mu\text{m}$  and 8 nm in thickness. All reagents were used without further purification. N,N-dimethylformamide (DMF) solvent was purchased from Sigma Aldrich, UK.

### 2.2. Preparation of polymer composite solutions

#### 2.2.1. Graphene nanoplatelets (GNPs) suspensions

Graphene nanoplatelets with weight percentage of 5% in DMF were sonicated in an ice bath using an ultrasound sonifier (Branson sonifier 250) at a power output of 60% for 4 h. It should be noted that DMF was used as the processing solvent because of its high boiling point ( $\sim 153^\circ\text{C}$ ). Using DMF in gyration gives more time for formation of fibres and hence stretching of the fibres is facilitated.

#### 2.2.2. Polymer solution containing GNPs

Three different TPU concentrations (15 wt%, 20 wt% and 25 wt%) were prepared using DMF in air tight bottle and magnetically stirred for 50 h at ambient temperature ( $\sim 20^\circ\text{C}$ ). The prepared solutions were used to make polymer solutions containing GNPs. For this purpose, the above prepared GNP suspensions were ultrasonically shaken in an ice bath using an ultrasound sonifier (Branson sonifier 250) at a power output of 60% for 30 min. Then from the suspension of GNPs, few millilitres of solution (5 wt%) was added into each polymer solution. The resulting polymer-graphene solutions were magnetically stirred for 5 h at ambient temperature. The final solutions were again ultrasonically shaken for 30 min before being used in pressurised gyration at ambient temperature.

Similarly, three different PR concentrations (10 wt%, 15 wt% and 25 wt%) were prepared using DMF in air tight bottles and magnetically stirred for 50 h at ambient temperature. Separately, 10 wt% PVP was prepared in de-ionised water in an air tight bottle at ambient temperature. Then from the suspension of GNPs, a few millilitres solution of (5 wt%) was added into 10 wt% PVP solution and stirred for 3 h. The PVP was added to modify and improve the rheological behaviour of the spinning solution. Finally, this solution was added to each PR polymer solution and ultrasonically treated for 30 min before being used in pressurised gyration.

### 2.3. Pressurised gyration

The gyration system consists of a rotary aluminium cylindrical vessel containing 20 small round orifices equally spaced on the vessel surface. The dimensions of the vessel and orifices are 60 mm in diameter with a height of 25 mm, and 0.5 mm in diameter located at the same vessel height, respectively. One end of the vessel was joined to pressurised gas with a rotary joint. A regulator controlled the flow of gas into the vessel. The bottom end of the vessel was connected to a DC motor, which can produce variable speeds up to 36000 rpm. In order to investigate the nanofibre size

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