



# Flow-induced crystallization of polypropylene in the presence of graphene nanoplatelets and relevant mechanical properties in nanocomposite fibres



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## ABSTRACT

Flow-induced crystallization of polypropylene (PP) in the presence of graphene nanoplatelets (GnPs) was experimentally observed for the first time. Monofilaments of PP and PP/GnPs in the presence and absence of polypropylene grafted maleic anhydride (PP-g-MA) as a compatibilizer were produced via masterbatch dilution technique. Morphology, crystalline and super molecular structures, thermal and mechanical characteristics of the nanocomposite fibres were investigated by means of SEM, WAXD, DSC, DMTA, TGA, polarized light microscopy and tensile testing. The fractured cross-section images indicated a reasonably good GnPs dispersion in the compatibilized nanocomposite fibres since a few large GnPs aggregates were observed. Based on DSC, XRD, and polarizing optical microscopy results, it can say that GnPs acted as a nucleating agent and increased the crystallization temperature and simultaneously acted as a physical barrier, reducing crystallite growth of PP. The introduction of PP-g-MA into PP/GnPs nanocomposite fibre increased storage modulus. Upon inclusion of PP-g-MA, the most significant improvement of 48% was observed for tensile modulus with the incorporation of 0.5 wt.% GnPs. The observed results suggested that the presence of PP-g-MA changed the internal structure and morphology of PP/GnPs nanocomposite fibres rather than improving the interface of PP/GnPs. We concluded that using extensional flow and having proper dispersion of GnPs, flow-induced crystallization could be occurred in PP/GnPs nanocomposite fibres. About 20% increase in crystallinity was found for the compatibilized PP/GnPs nanocomposite fibres as compared to that of the virgin PP.

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## 1. Introduction

Graphene, or mono-layer graphite, is a single atom thick sheet of 2-D bonded carbon atoms, which are tightly packed in a hexagonal crystalline structure which is considered a basic structural unit for some carbon allotropes of various dimensionalities [1–3]. Graphene nanoplatelets (GnPs) are platelet-like graphite nanocrystals with multigraphene layers. In recent years, graphene/polymer nanocomposites have attracted attention as a promising candidate to create new materials with novel properties due to their excellent mechanical, electrical and thermal properties. Graphene/polymer nanocomposites have shown potential applications in the fields of electronic, aerospace, automobile, defense industries, green technology, etc. [1,4–8].

Polypropylene (PP) is an engineering thermoplastic polymer with good physical and mechanical properties, chemical resistivity, easy processing, and low cost. During the last decade, PP/graphene nanocomposites were studied extensively [2,9–16]. These researches showed that the properties of nanocomposites, such as mechanical properties, are dependent on the state of dispersion of graphene in the matrix and loading amount of graphene. Inhomogeneous GnPs dispersion and the presence of large aggregations result in significant deterioration of the mechanical properties of final nanocomposites. Thus, achieving a homogenous dispersion of GnPs in the polymer matrix has an important role to avoid the deterioration of the mechanical properties of the PP/GnPs nanocomposites.

Although GnPs are non-polar materials, they could contain a few functional groups depending on their preparation methods. These functional groups could be bonded to the polymer matrix to achieve proper interaction between GnPs and polymer matrix. Additives used to improve interfacial bonding between GnPs and

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the polymer matrix are referred to as coupling agent or compatibilizer. A large number of scientific literatures have been focused on the preparation of PP nanocomposites with different nanofillers using various compatibilizer agents [17–21]. Among them, polypropylene-grafted-maleic anhydride (PP-g-MA) is the most convenient compatibilizer to be used. However, little effort is devoted to investigate the PP/GnPs nanocomposites in the presence of a compatibilizer. Duguay et al. [15,16] have found that for PP/GnPs composites containing a compatibilizer, tensile modulus increased while elongation at break and impact properties deteriorated. Moreover, they concluded that the addition of compatibilizer dramatically enhanced GnPs dispersion within GnPs-filled PP composites. Roh et al. [22] proved that for PP/graphite nanoplatelets composites containing 10 wt.% of PP-g-MA, the mechanical properties improved as compared to PP/graphite nanoplatelets composites without PP-g-MA. Recently, Pedrazzoli and Pegoretti [23] investigated possibility to improve the adhesion between E-glass fibres and PP by dispersing GnPs in the polymer matrix. They found that the strength at PP/glass fibre interface can be remarkably increased by the addition of GnPs, and that the improvement was further enhanced when GnPs were used in combination with PP-g-MA.

In typical thermoplastic polymer processing including fibre melt spinning, injection moulding and film blowing, the application of complex flow fields can strongly enhance the crystallization rate. This phenomenon is usually known as flow-induced crystallization. Due to the effect of flow on the final morphology and properties which will have great influence on the mechanical properties of polymer, this has been widely investigated and reported in the literatures [24–28]. Most of the studies on the flow-induced crystallization have been done on the pure polymers. However, flow-induced crystallization in the presence of nanoparticles is much more complex than that in the pure polymer [29–36]. Since particles disturb the flow field, a significant effect of presence of particles on the flow-induced crystallization is expected. Numerical simulation study of flow-induced crystallization of polymer filled with rigid particles by Hwang et al. concluded that the presence of particles in polymer melts produce regions with highly oriented polymer chains between the spherical particles that are in close proximity. Moreover, the highly homogeneous molecular conformation in a particle-filled polymer directly affects flow-induced crystallization [37]. The nanoparticles can act as nucleation sites for heterogeneous crystallization. The number of heterogeneous sites available for nucleation is mainly affected by the concentration and dispersion of particles in the polymer matrix. The combined effect of flow and addition of particles have been suggested to yield a synergistic increase in the number of active nuclei, raising crystallization rate further than their individual contribution [31].

This study was conducted to understand if GnPs dispersion is successfully accomplished, whether this results in flow-induced crystallization under extensional flow and how this ultimately influences the mechanical properties of the final product. Moreover, morphology, microstructure, thermal and mechanical properties of PP/GnPs nanocomposite fibres in the presence of PP-g-MA was investigated because no report on the properties of PP/GnPs nanocomposite fibres upon addition of a compatibilizer can be found in the open scientific literature.

## 2. Materials

A commercial fibre grade of isotactic polypropylene, known as 512P (Sabic Co., Saudi Arabia), with a molecular weight of 186,000 g/mol and a melt flow index of 25 g/10 min was used. This material contains the usual synergistic combination of

antioxidants that are found in the commercial granules. Polypropylene-grafted-maleic anhydride, PP-g-MA, was used to enhance the interface interaction between PP and GnPs, because as quoted by supplier, GnPs have various functional groups such as carboxyl and hydroxyl groups which are available at the GnPs edges and be able to react with PP-g-MA. A commercial PP-g-MA with a trade name of Polybond® 3150 (Chemtura Co., USA) was used as a compatibilizer. The grafting level of compatibilizer was 0.5 wt.% as given by the supplier. A masterbatch of PP/GnPs, containing 10 wt.% graphene nanoplatelets (GnPs), was obtained from Ovation Polymers, USA. According to supplier, the GnPs used was provided by XG Sciences Inc., East Lansing, USA, which have the average thickness and mean diameter of 6–8 nm and 5 µm, respectively.

## 3. Methods

### 3.1. Compounding conditions

All nanocomposite masterbatches were prepared in 400 g batches and all constituents were added to the twin-screw simultaneously. The PP/GnPs and PP/MA/GnPs compounds with the desired GnPs content were obtained by melt blending in a ZSK-25 co-rotating twin-screw extruder (Coperion Werner & Pfleiderer) with length/diameter ratio of 40. The temperature of the extruder zones from the feed stock to the head were 175, 185, 195, and 200 °C and the screw speed was 250 rpm. The extruded strands were quenched in water and then pelletized for melt-spinning process. PP homopolymer granule was also extruded under the same conditions to have similar extruded raw material for melt spinning. The designated labels and compositions of all compounded materials are listed in Table 1.

### 3.2. Melt spinning of PP/GnPs nanocomposite fibres

The granules were dried in a circulating air oven for 24 h at 80 °C prior to melt-spinning. The melt-spinning process was performed using a laboratory melt-spinning machine. A single-hole spinneret with a diameter of 1000 µm was used for the production of fibres. The temperatures of heating zones of extruder were set at 195, 215, 230, 235, and 240 °C and take-up speed was 100 m/min.

### 3.3. Fibre testing and characterization

The monofilament tensile fractured cross-section and cryofractured cross-section morphologies were studied with two field emission scanning electron microscopes (SIGMA VP, Zeiss, Germany and FESEM, Hitachi S4160, Japan). Cryofractured cross-section samples were prepared by freeze cutting in liquid nitrogen and all fibre samples were coated with gold before being examined with the microscope at an acceleration voltage of 20 kV.

**Table 1**

Sample designation and formulation of the compounded masterbatch granules and their corresponding fibres.

Sample designation	PP content, wt.%	GnP content, wt.%	PP-g-MA content, wt.%
Virgin PP	100	–	–
PP/MA	99	–	1
PP/G0.1	99.9	0.1	–
PP/G0.5	99.5	0.5	–
PP/MA/G0.1	98.9	0.1	1
PP/MA/G0.5	98.5	0.5	1

The number for G shows the weight percentage of graphene nanoplatelets in the fibre.

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