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Influence of processing conditions on the mechanical behavior of MWCNT reinforced thermoplastic nanocomposites

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Abstract

The influence of the processing conditions and MWCNT content on the mechanical properties of PA6,6-based nanocomposites are investigated. In addition to the composition of the composites, the impact of manufacturing conditions such as dilution mechanism, twin-screw extruder mixing specifications, and injection molding parameters on the behavior of the nanocomposites are evaluated. Results show that while the increase in the content of MWCNTs can lead to 40.0 % enhancement in the mechanical properties, changing the processing parameters varies the values by 30.0 % in the same content. The mechanisms involved in the modulation of the nanocomposites properties are also discussed.

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1. Introduction

Recent advancements in the realm of science and technology have created new possibilities and opportunities for different industrial applications. Among these novel possibilities, carbon nanotubes (CNTs) with their various extraordinary properties have attracted unprecedented interest of researchers and scientists from all over the world in the last two decades. Since their discovery in 1991, significant research has been underway to study their unique properties [1-3]. In fact, because of their exceptional properties such as mechanical, electrical, thermal, etc. features, composites reinforced with CNTs are being considered as the replacement to many conventional materials in various industrial applications including automotive, aerospace, sport equipment, energy, and infrastructure sectors [4]. However, despite their increasing applications, lack of extensive research that is mainly targeting tailored manufacturing of these materials especially in the industrial scales can be noted easily. In fact, there are just a few papers that have focused on the behavior of the CNT reinforced nanocomposites manufactured through industrially viable techniques [5-8]. It

is also noteworthy to mention that the main focus of most of these few papers were the electrical properties of the polymeric nanocomposites.

Polyamide or Nylon 6,6 (PA 6,6) is one of the most well-known engineering polymers with different applications in various industrial sections especially automotive industry. Some prominent properties such as good mechanical behavior, fair heat and fatigue resistances, low-temperature behavior, resistance to oils, fair cost, stability in the manufacturing processes, etc. have made PA 6,6 and reinforced PA6,6 one of the most consumed polymers in the world. In recent years, regardless of their particular susceptibility to humidity, they are replacing metal components in various applications where they are offering better mechanical and chemical performances with less weight [9].

Injection moulding of components from thermoplastic polymers is a preferable and established industrial production method because of its considerable advantages in cost and time. However, the involved parameters in this process can notably influence the properties of polymeric composites. Nanocomposites reinforced with Multi Walled Carbon Nanotubes (MWCNTs) are considerably influenced by the

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defined state of the polymer and nanofillers during both melt and cooling states. In fact, the properties of the nanocomposites are highly influenced by the distribution, dispersion, alignment, and the interfacial properties of MWCNTs in the polymer system. In other words, parameters such as injection speed, injection pressure, melting temperature and mould temperature define the quality of the nanocomposites [6,9,10]. Moreover, it should be noted that based on the specific targeted properties, the state of MWCNTs in the nanocomposites should be tailored differently. However, despite their soaring applications in different industries, only a few papers have focused on the sensitivity of the final behaviors as a result of the different manufacturing processes.

The purpose of this study is to investigate of the influence of the manufacturing process parameters on the mechanical properties of polymeric nanocomposites. PA 6,6-based nanocomposite specimens with different contents of MWCNTs were prepared via different methods. Subsequently, the prepared specimens were characterized using uniaxial tensile experiments and the influence of the processing parameters i.e. dilution and injection molding parameters on the mechanical behavior were studied in detail. In order to find the influential mechanisms on the variations of the observed mechanical properties, rheological analyses and scanning electron microscopy (SEM) were employed.

2. Experiments

2.1. Materials

The multi-walled carbon nanotubes used in this study are catalytic chemical vapor deposition produced thin MWCNTs (NC 7000 $^{\rm TM}$) by Nanocyl SA, Belgium, with the average aspect ratio of 67 (d_{ave} =10.4 nm) [5]. The PA 6,6 based masterbatch containing 15.0 wt. % of the aforementioned MWCNTs (according to the supplier Nanocyl SA) were selected in the manufacturing processes to acquire the desired contents in the nanocomposites. The masterbatch was selected especially because of the application of industrial MWCNTs within the polymer, and viability for mass production. In addition, Altech PA 6,6 (ALBIS Plastic GmbH) was selected to mix with the masterbatch in the dilution process because of its high melt flow index which is assumed to facilitate the dispersion of the nano fillers in the matrix (Fig. 1. (a) and (b)).

2.2. Melt mixing

Melt compounding is a well-stablished and preferred method in the industrial scale production of nanocomposites because of its favorable features such as creating considerably less pollutant residuals, mass production, high speed, and less cost. This process consists of melting the selected polymer at high temperatures, and mixing it with the defined amount of nano additives through rotation and shear forces to reach the desirable content of the fillers in the matrix. However, the quality of the products depends on several parameters such as polymer characteristics, compatibility of the nanofillers and the matrix, interface behavior, etc. In order to study the

influence of the dilution process on the mechanical properties of the nanocomposites two main approaches were compared. The first dilution method, which is called "direct method" in this paper from now on, is hand-mixing the master-batch and the neat polymer, which is followed by the subsequent melt mixing in the barrel of the injection unit of the injection molding machine. In fact, in this process, the two different pellets are melted, and mixed simultaneously under heat, rotation and shear force originating from the hot rotating screw just before injection into the mold. This method has especially been favorable in mixing different colorants with polymers. The second method is the application of twin-screw extruder before injection molding to mix the two different polymeric pellets, and preparing the new compounds.

The conical counter rotating twin-screw extruder (HAAKETM Rheomex CTW, Φ =31.8/20 mm rear/front, L=300 mm) was selected to perform the melt mixing process, since it is assumed that the high shear force would be beneficial for the de-agglomeration of the carbon nanotubes. Prior to feeding the materials into the extruder, the neat polymer and master batch pellets were weighted, and hand mixed to the desired contents, namely 0.5, 1.0, 3.0, 5.0, and 6.0 wt. % of MWCNTs. The mixtures were dried before and after the twin-screw extruder process for respectively 4 and 6 hours at 80 °C in order to exclude any influence of humidity for either mixing or injection molding. The temperature distribution through the five zones (from feed section to die) kept from 265 to 280 °C, with the average temperature of 275 °C. The melting screw speeds were 15, 25, and 50 rpm, with residence times of 12, 8, and 4 min, respectively (Fig. 1. (c) and (d)).

2.3. Injection molding

Injection molding of the nanocomposite specimens was performed on Ferromatik, Milacron following the instructions of ISO 294-1 standard. The geometry of the cavity of the mold was dog-bone designed based on ISO 527-2 2012 standard. A series of experiments were conducted using a two level, four factor factorial design to investigate the influence of the four considered parameters on the mechanical properties of the nanocomposites. The four factors of injection velocity, melt temperature, mould temperature, and holding pressure were variated between the predefined minimum and maximum values through 16 experiments (see Table.1).



Fig. 1. (a) PA 6,6, (b) MWCNT filled masterbatch, (c) hand mixed masterbatch/ PA6,6, and (d) nanocomposite product of twin screw extruder.

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