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Material properties

Optimization of mechanical properties of polypropylene/talc/graphene composites using response surface methodology

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ABSTRACT

This paper investigated the reinforcing effects of a hybrid filler, including talc and exfoliated graphene nanoplatelets (xGnPs), in polypropylene (PP) composites. In order to increase the interphase adhesion, maleic anhydride grafted polypropylene (MAPP) was added as a compatibilizing agent to the PP/talc/xGnP composites. The experiments were designed according to response surface methodology (RSM) to optimize the effects of three variable parameters, namely talc, MAPP and xGnP, on the mechanical properties. In the sample preparation, three levels of filler loading were used for talc (0, 15, 30 wt%), xGnP (0, 0.75, 1.5 wt%) and MAPP (0, 2, 4 wt%). From the analysis of variance (ANOVA), it was found that the talc and xGnP play a significant role in the mechanical properties and morphology of the composites, as proven by scanning electron microscopy (SEM) and differential scanning calorimeter (DSC). In order to simultaneously maximize these mechanical properties, the desirable values of the additives were predicted to be 30 wt% for talc, 4 wt% for MAPP and 0.69 wt% for xGnP. The obtained normal probability plots indicated good agreement between the experimental results and those predicted by the RSM models.

1. Introduction

Polypropylene (PP) is one of the most widely used thermoplastics and has been employed in the construction, automotive, packaging, cable insulation and household goods industries [1–3]. However, PP has some drawbacks such as the development of static electricity, low impact toughness and poor adhesion of hydrophilic reactive groups in PP chains [4]. Therefore, there is much focus on PP-based composites to improve the mechanical and thermal properties.

The utilization of fillers from various sources added to the PP composites has been an accepted route to achieve enhancement in mechanical properties, cost saving possibilities, or a combination of both [5,6]. The presence of talc in thermoplastics usually results in improved tensile and flexural properties, but it decreases impact strength [4,7]. This filler also facilitates the shaping of PP by reducing and homogenizing the molding shrinkage. This is important for jointless connections and zero-gap parts such as automotive bumpers [8]. The concentration of talc as a reinforcement in plastics is usually between 20 and 40 wt% [9]. The high density of talc (~2.5–2.7 g/cm^3) increases the weight to volume ratio of the composite when applied in a significant amount [10]. To increase the interaction between the talc and PP chains, a compatibilizer or surface modification of particles is applied. Maleic anhydride grafted polypropylene (MAPP) has been used as a compatibilizer to enhance the adhesion between talc and PP matrix and promote intercalated structure.

In recent years, researchers have focused their interest on polymers reinforced with nanosize materials, which represents an alternative to conventional filled polymers. The number of carbon-based nanoreinforcement materials is growing; they range from carbon nanotubes, carbon nanofibers and fullerenes to graphene [11]. Exfoliated graphene nanoplatelets (xGnPs) have attracted great attention in recent years owing to their exceptional mechanical, thermal, optical and electrical properties [12]. To date,

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most work in the area of graphene-based polymer nanocomposites has focused on property modification using graphene [13–15]. This emphasis can be partly attributed to the intrinsic properties of xGnP, which result in remarkable property improvements at low graphene concentrations [16]. It was also reported that the improvements in the mechanical and electrical properties of xGnP based polymer composites are much greater than those of nanoclay or other carbon filler-based polymer composites [17,18].

Response surface methodology (RSM) is a collection of useful statistical and mathematical techniques for developing, improving and optimizing processes [19]. RSM also provides more analysis of the interactions between variables than other experimental design methods. Therefore, utilizing RSM in optimization gives a more accurate and complete data with a minimal number of experiments [11].

To achieve high performance and commercial success for polymer nanocomposites with low nanofiller content and the availability of cost-effective nanofiller with suitable inherent properties, the optimization of the fabricating process is the first key factor to be considered. In this study, the RSM method is used to drive formulas that describe the relations between the mechanical properties of the PP/talc/xGnP nanocomposites. Then, the results attained by the formulas were compared with the experimental results for investigating the accuracy of the formulas and also calculating the possible errors. Based on our literature search, there is no information on the mechanical properties of PP/talc/xGnP composites.

2. Experimental

2.1. Materials

The PP material used in this study was a commercial polymer (PP-V30S trade name) with a melt flow index (MFI) of 18 g/10 min and a density of 0.918 g/cm^3, which was supplied by the Arak Petrochemical Co. (Iran). The maleic anhydride grafted polypropylene (MAPP, 2 wt% grafting level) with an MFI of 10 g/10 min and a density of 0.91 g/cm^3 was supplied by the Karangin Co. (Iran) and used as a compatibilizer. The graphene used in this study was xGnP-C750-grade material produced by XG science (USA). The xGnP particles typically consist of aggregates of sub-micron platelets that have a particle diameter of less than 2 μm, a typical particle thickness of a few nanometers, and an average surface area about 750 m^2/g. The xGnP-C750 nano-platelets have naturally occurring functional groups like ethers, carboxyls and hydroxyls (totally containing 8 wt% of oxygen). The talc (Mg-silicate), with the trade name of Microtalc IT EXTRA and a mean particle size of 1.7 μm, was obtained from the Norwegian Talc Co. (Norway). All materials were used as received without further purification.

2.2. Preparation of sample

The materials were compounded by melt-blending using a Brabender internal mixer at 60 rpm and 180 °C for 10 min. The samples for tests were prepared by hot-press molding using a square steel mold (250 × 250 mm^2) at a temperature of 190 °C. The blends were first added to the cold mold. The platens were preheated to 190 °C and a minimum pressure was applied during the preheating step to maintain contact between the platens and mold. The pressure was then increased slowly to 2.5 MPa in 2 min and held at this pressure for an additional 5 min. The mold was then removed from the hot press and cooled to room temperature in a separate cold press under the same pressure (2.5 MPa).

2.3. Design of experiments

A common RSM design, which is also known as the Box-Behnken design, was employed in the present work by using the statistical software Minitab® 17. According to the Box-Behnken design, three independent variables namely talc, MAPP and xGnP were considered at three coded levels (−1, 0, +1) to develop the experimental design, as given in Table 1. The experiment consisted of 15 runs with an average of three replicates for each (Table 2). The experimental sequence was randomized in order to minimize the effects of the uncontrolled parameters.

2.4. Characterization

The tensile properties were determined using a Zwick/Roell machine (model z100) at a crosshead speed of 5 mm/min, according to ISO 527-1. Notched Izod impact tests were conducted with a Santam pendulum impact tester (model SIT-20D), according to ISO 180. The morphology of the samples was studied by scanning electron microscopy (SEM) (model WEGA-II TESCAN) from the fracture surfaces of the tensile tests. The samples were coated with a thin film (15 nm) of gold to avoid electrical charge accumulation during the examination and then analyzed at an accelerating voltage of 20 kV. Differential scanning calorimetry (DSC) was performed by a Netzsch DCS 200 F3 Maia device under nitrogen atmosphere to investigate the thermal behavior of the samples. The DCS procedure consisted of three steps: first, the samples were heated from 25 to 200 °C at a rate of 10 °C/min; then they were held at this temperature for 5 min to eliminate the thermal history and cooled to 25 °C at a cooling rate of 10 °C/min. Finally, they were reheated to 200 °C at a rate of 10 °C/min.

3. Results and discussion

3.1. Mechanical properties

The mechanical properties of PP/talc/xGnP composites were analyzed statistically using an analysis of variance (ANOVA) in order to identify the effect of the three parameters namely talc, MAPP and xGnP on the tensile strength, tensile modulus and impact strength. Table 3 shows the results of the ANOVA analysis for the tensile strength. The ANOVA Table shows the sum of the squares (SS) and mean square (MS) of each parameter where the P-value and F-ratio are defined as the ratio of the respective mean square effect and the mean square error. For a 95% confidence level, an effect is usually considered significant when the p-value is less than 0.05. According to the F-ratio and P-values shown in Table 3, it can be seen that the effects of talc, MAPP and xGnP, as well as the interactions effects of xGnP × xGnP and talc × MAPP, are statistically significant for the tensile strength of the PP/talc/xGnP composite. A polynomial model with statistical significance can be generated from a combination of estimates for the parameters and the ANOVA results for the tensile strength. This model, quantitatively clarifying the effects of parameters with statistical significance in coded unit (i.e., −1, 0, +1), is presented as follows:

Table 1
Variables in Box-Behnken experimental design.

Parameters	Levels used		
	Low (−1)	Middle (0)	High (+1)
Talc (wt%)	0	15	30
MAPP (wt%)	0	2	4
xGnP (wt%)	0	0.75	1.5