



# Electron-induced reactive processing of polyamide 6/polypropylene blends: Morphology and properties

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## ABSTRACT

Electron-induced reactive processing (EIREP) recently has appeared as an advanced processing technology to modify several polymer compounds at melt state by its spatial and temporal precise energy input. In the present work, an attempt was made to prepare a compatible blend from polar polyamide 6 (PA6) and non-polar polypropylene (PP) using EIREP without any chemical crosslinking and chemical additives. EIREP modified blends were prepared at different doses (5, 10 and 20 kGy) and dose per revolutions (3, 6 and 12 kGy/rev) while keeping constant the values of mass ratio (PA6/PP 50/50 w/w), electron energy (1.5 MeV), rotor speed (60 rpm) and temperature (230 °C). EIREP process parameters-morphology-property relationship of this blend was studied. Interestingly, co-continuous morphology of the blend was converted to droplet-matrix morphology after EIREP whether PA6 was continuous phase. Influence of dose and dose per revolution on the DMT modulus (Derjaguin-Muller-Toporov) at different phases of the blends was also measured and analyzed using advanced PeakForce Quantitative Nanomechanical Mapping atomic force microscopy (PF QNM AFM) technique. Improved nano- and macroscopic properties of EIREP modified blends were originated from the properties of the continuous matrix phase, electron-induced branching and also from the reduction of domain size of PP phase with increased dose.

## 1. Introduction

Melt blending of polymers is an economic and efficient method to design new high performance polymeric materials [1,2]. However, most of the polymer blends are thermodynamically immiscible due to their difference in chemical structure and polarity, and exhibited poor mechanical properties, poor interfacial adhesion, unstable morphology, etc. [3,4]. It is well known that properties of polymer blends depend on morphology, interfacial adhesion, interfacial tension and also on the properties of blend components [5–9]. Generally, several types of compatibilizer and also recently nanoparticles are being used to improve interfacial adhesion and morphological stability of immiscible polymer blends [10,11]. Recently, it was shown that electron-induced reactive processing (EIREP) is also an efficient technique for stabilization of morphology and interfacial adhesion of immiscible polymer blends [12].

EIREP is an advanced processing technology developed at Leibniz Institute of Polymer Research Dresden for reactive compounding of various polymeric materials. The properties and morphology of the polymers can be controlled by using spatial and temporal precise

energy input from high energy electrons [13]. The electron energy during EIREP influences the penetration depth of electrons within the polymer melt as well as the ratio of reaction volume (treatment zone) to total mixing volume. Finally, the whole volume of polymer melt can be modified [13]. Several polypropylenes (PP) based compounds and thermoplastic vulcanizates have been developed using this technique [14–19]. Recently, high performance polymer blends were developed using immiscible polypropylene/epoxidized natural rubber and polyamide 6/fluoroelastomer systems [12,20]. In this article, an attempt was made to prepare a PA6/PP blend by EIREP without any use of chemical compatibilizer.

Polyamide 6 (PA6) is a high performance polymer having good mechanical properties, chemical resistance, low friction, high melting point, etc. [21]. On the other hand, PP is a low cost polymer having good processability, mechanical properties, etc. [22]. However, PA6 and PP are inherent immiscible polymers due to differences of their chemical structure and polarity. There are several reports on compatibilization of PA6/PP blends using chemical additives [23–25]. In addition, electron-beam (EB) processing was also performed to develop PA6/PP blends [26]. Generally, EB processing was done at ambient

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temperature. In the case of EB processing of immiscible polymer blend, a gap at the interface is expected and generally a reactive agent is needed to overcome this gap before EB processing. Such type of EB processing of PA6/PP blends in the presence of a reactive agent is reported in the literature [27]. In the present research, EB processing was performed at melt state (EIREP), where maximum mobility of PA6 and PP chain segments were obtained and such interfacial gap could be reduced in the melt-state. In addition, dose values applied to the PA6/PP melt mixture are less than the gel dose of PA6 and also less than the dose where PP dominates to degrade [28]. Therefore, branching of both phases of the blend was expected. Finally, it has to be mentioned that EIREP modified PA6/PP blends can be recycled and also be used for extrusion or injection moulding as there is no crosslinking in the blend system.

The objective of the present work is to prepare PA6/PP blends by EIREP without any use of chemical additives. Morphology, rheology, mechanical and dynamical mechanical properties were measured and analyzed as function of applied dose (absorbed energy per unit of mass). PeakForce Quantitative Nanomechanical Mapping (PF-QNM) is an advanced atomic force microscopy (AFM) technique to obtain mechanical properties of several polymeric materials at nanoscale [29]. However, it is a challenge to obtain quantitative mechanical properties at different phases of polymer blends at nanoscale. In this present research, QNM AFM was explored to understand the effect of nanomechanical properties of EIREP modified and unmodified PA6/PP blends.

## 2. Experimental

### 2.1. Materials

PA6 (Ultradim B27E) having density 1.13 g/cm<sup>3</sup>, melting temperature 220 °C, number average molecular mass 68,500 g/mol and melt flow rate 140 g/10 min (275 °C and 5 kg) was kindly supplied by BASF (Germany). Isotactic PP (HG455FB) having density 0.98 g/cm<sup>3</sup>, melting temperature 165 °C, number average molecular mass 84,800 g/mol and melt flow rate 23 g/10 min (230 °C and 2.16 kg) was obtained from Borealis (Düsseldorf, Germany).

### 2.2. Preparation of PA6/PP blends using electron-induced reactive processing

PA6 granules were dried in a vacuum oven for 24 h at 80 °C to remove absorbed moisture before mixing. In order to modify the molten PA6-PP mixture, a discontinuous EIREP set-up was used. This set-up consists of an electron accelerator ELV-2 (BINP Novosibirsk, Russia) which was coupled to a Haake internal mixer (Fig. 1). EIREP modified blends were prepared under nitrogen atmosphere at varying doses (5, 10 and 20 kGy), electron currents (0.35, 0.70 and 1.40 mA), and dose per revolutions (3, 6 and 12 kGy) while the mass ratio (PA6/PP 50/50 w/w), mixing temperature (230 °C), rotor speed (60 rpm), electron energy (1.5 MeV), and electron exposure time (15 s) were kept constant.

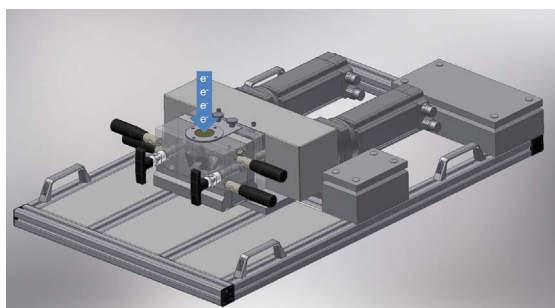


Fig. 1. Scheme of discontinuous EIREP setup.

During EIREP, nitrogen atmosphere was used in order to avoid any change of the polarity of both polymers as well as to reduce thermal and electron induced degradation of PP. Total mixing time was 5 min. Control non-reactive 50/50 (w/w) PA6-PP blend composition was also prepared without applying any dose. Above dose values were selected on the basis of gel content in PA6 and EB induced degradation of PP. It was found that up to 20 kGy dose, PA6 have no gel. From our recent study, it was revealed that pure PP has small degradation up to 20 kGy dose [30].

### 2.3. Sample preparation

Test samples were prepared using a vacuum compression moulding machine (Rucks Maschinenbau, Germany) at a temperature of 230 °C and a pressure of 6 MPa for 3 min.

## 3. Testing procedure

### 3.1. Gel content

The gel content of PA6 was estimated from solvent extraction method using formic acid as a solvent. After extraction in formic acid for 16 h at 30 °C, PA6 was dried in a vacuum oven and weighed. Gel value ( $X_g$ ) in percentage was calculated from the following equation [19]:

$$X_g = \frac{m_1}{m_0} \times 100 \quad (1)$$

where  $m_0$  and  $m_1$  are the mass before and after extraction, respectively. It was found that PA6 have no gel up to dose values of 20 kGy.

### 3.2. Mechanical testing

The tensile tests were performed using Zwick Universal Testing Machine (Zwick 8195.04) according to ISO 527-2/S2/50 specification at room temperature. The cross-head speed was kept at 50 mm/min. The tensile strength and Young's modulus values reported here are the average of at least three samples from the same batch.

### 3.3. Dynamical mechanical analysis

The dynamic mechanical properties were measured using an Eplexor 2000N DMTA (version 8.373 h) in tension mode at a constant frequency of 1 Hz, at a heating rate of 2 K/min, and 0.2% strain over a temperature range of  $-100$  °C to 100 °C. Storage modulus was measured and analyzed as a function of temperature.

### 3.4. Melt rheology

The melt rheological measurements were performed by using an ARES rheometer (Rheometrics Scientific, New Castle, USA) in a parallel plate geometry having diameter of 25 mm. Frequency sweep experiments were conducted at 230 °C temperature, 0.1–100 rad/s frequency range, and 1% of constant strain in nitrogen atmosphere. The absolute value of complex viscosity, storage modulus, loss modulus and phase angle were measured as a function of frequency.

### 3.5. Scanning electron microscopy (SEM)

The morphology of the blends were captured using LEO 435 VP Ultra plus Scanning Electron Microscopy (Carl Zeiss SMT, Germany) with 10 kV accelerating voltage. Before SEM analysis, samples were cryo-microtomed (Leica Ultra-microtome) at  $-100$  °C and also sputter-coated with platinum for 20 s. For each blend sample, at least ten images were captured and analyzed.

From the SEM images, the number average diameter ( $\langle d_n \rangle$ ) and

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