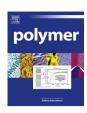
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Novel nanostructured polyamide 6/fluoroelastomer thermoplastic elastomeric blends: Influence of interaction and morphology on physical properties



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ABSTRACT

Novel polyamide 6 (PA6)/fluoroelastomer nanostructured thermoplastic elastomeric blends were developed in the present work. The influence of interaction between the components and morphology on physical properties of the blends was analyzed. Scanning electron microscopy and atomic force microscopy studies, solubility and theoretical analysis of complex modulus clearly indicated that PA6 was the continuous matrix in which fluorocarbon elastomer was present in nanoscale. Low torque ratio (0.34) of rubber/plastic, high mixing speed and long mixing time had an important role in developing the nanostructured morphology of the blend. Tensile strength of the thermoplastic elastomer was about 39.0 MPa which was much higher than that reported earlier and showed significant improvement with increasing PA6 content. Large shifting of the glass transition temperature of the rubber and the plastic phases towards the lower temperature compared to those pristine polymers was also observed. The above properties were explained with the help of interaction between the components and morphology.

1. Introduction

Thermoplastic elastomers (TPEs) have appeared as one of the most important classes of specialty materials having immense potential for industrial revolution. They have unique and extraordinary properties such as excellent physico-mechanical properties, chemical resistance, wider processing windows, ability to bond with multiple thermoplastics, light weight and excellent priceperformance ratio, all of which make these an ideal candidate for enormous industrial applications [1-4]. Polyamide based thermoplastic elastomers are reported to have great importance because of their excellent strength, stiffness, low friction, high melting point, and chemical and wear resistance [5]. Thermoplastic elastomers based on polyamides, prepared by melt mixing process are expected to have excellent heat and oil resistance [6]. Several studies of different properties of blends of polyamide and rubbers are available in the literature. Crisenza et al. studied direct 3D visualization of the phase-separated morphology of thermoplastic elastomers from chlorinated polyethylene/nylon terpolyamide blends [7]. Jha and Bhowmick investigated the reactive nylon-6/acrylate thermoplastic elastomeric blends and their interaction on

mechanical and dynamic mechanical properties [8]. Oderkerk and Groeninckx reported the morphology development by reactive compatibilization of nylon-6/EPDM blends with a high amount of rubber fraction and extensively studied the effect of viscosity ratio of rubber/thermoplastic on the blend morphology [9].

Coran and Patel extensively investigated the thermoplastic elastomers from various rubber—plastic blends and correlated the properties with morphology, surface energy mismatch and crystallinity of the matrix phase of the blends [10,11]. Few authors investigated the interaction between immiscible polymers through the addition of reactive components [12–14]. Mechanical properties, dynamic response and interaction between components (both reactive and non-reactive systems) of rubber—plastic blends are main concern with the rubber modified plastics systems. Many researchers reported non-covalent bonding interaction in polymer blends (mainly H-bonding interaction) through infrared spectroscopic analysis [15–18].

Among different classes of thermoplastic elastomers, blends of rubbers and plastics are gaining importance because the required property can be easily monitored by varying the ratio of the constituents, viscosity of the components and also by incorporation of additives like compatibilizer, crosslinking agents, fillers, etc. Even though many polyamide based TPEs are commercially available, development of polyamide-fluoroelastomer TPEs has not been reported in literature. Also, polyamide shows interesting processing

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problems and its blends with rubber without compatibilizer exhibit difficulty in processing and poor mechanical characteristics.

The present investigation reports our observations on the blends of polyamide 6 and fluoroelastomer by melt-blending process. The influence of interaction and blend ratio on dynamic mechanical and mechanical properties was investigated. FTIR study on H-bonding interaction between polyamide 6 and fluoroelastomer was highlighted. The molecular weight of polyamide is one of the most important parameters governing the melt viscosity and hence processing characteristics, interfacial adhesion and morphology of the blend. The rubber particle size in rubber toughened plastics can be reduced by an order of magnitude by increasing the molecular weight of polyamide keeping other process conditions constant [19–22]. Our objective is to develop a thermoplastic elastomeric blend with nanodimensional elastomeric particles dispersed in a plastic matrix. To the best of our knowledge, this is the first time that a nanostructured polyamide based thermoplastic elastomeric blends has been prepared by melt blending process. Furthermore, the effects of nanostructured morphology on the physical properties of the blends are reported.

2. Experimental section

2.1. Materials

Polyamide 6 (tradename Akulon) in granular form (viscosity number 245 cm³/g, density 1130 kg/m³, melting point 220 °C) was supplied by DSM, Nederland. Viton A (a copolymer of vinylidene fluoride (VF2) and hexafluoropropylene (HFP), density 1810 kg m $^{-3}$ at 25 °C, 66% F, Mooney viscosity, ML $_{1+10}$ at 121 °C = 20) abbreviated as FKM was procured from DuPont Dow Elastomers, Geneva, Switzerland. The formulation of the mixes is given in Table 1. The molecular structure of polyamide 6 and FKM is given in Fig. 1

$2.2. \ Preparation \ of \ thermoplastic \ elastomeric \ blend \ compositions$

PA6 granules were dried in vacuum oven at 100 °C for 24 h before blending. The blends were compounded in batches of 50 g polymer in a Haake Rhecord internal mixer with roller type rotor. Blending was allowed to proceed for 7 min at a temperature of 240 °C and 100 rpm rotor speed. After 2 min melting of PA6 in the internal mixer, FKM was added and mixed for another 5 min under the same conditions. The changes of mixing torque and energy with time were recorded for each composition. After complete mixing in the internal mixer, the resulting blends were quickly removed and passed through a two-roll mill having close nip-gap at room temperature to make sheets and were cut into small pieces.

One blend composition $\{40\ PA6/60\ FKM\ (w/w)\}\$ which showed best thermoplastic elastomeric behavior was also processed in the following two ways:

- i. Similar procedure as above was followed except high viscosity PA6 was replaced by low viscosity PA6.
- ii. Mixing was allowed to proceed for 3.5 min at a temperature of 240 $^{\circ}\text{C}$ and 50 rpm rotor speed.

The procedure followed after complete mixing was same as before.

2.3. Molding of thermoplastics elastomers

Test specimens (1–1.5 mm thick) were prepared by means of micro-injection molding machine (Haake MiniJet II) at 260 $^{\circ}$ C cylinder temperature and 60 $^{\circ}$ C mold temperature. Injection pressure

Table 1 Formulation of mixes.

Sample composition	PA6	FKM	Comments
PA6	100 ^a	_	(Pure components)
FKM	_	100 ^a	
80 PA6/20 FKM (w/w)	80	20	(Variation of plastic-rubber
60 PA6/40 FKM (w/w)	60	40	ratio)
40 PA6/60 FKM (w/w)	40	60	
20 PA6/80 FKM (w/w)	20	80	

a Weight percent.

and holding pressure were 700 bar and 350 bar respectively. The injection and holding times were 5 s each.

2.4. Characterization

2.4.1. Solubility measurement

Approximately 0.5 g (accurately weighed) of each blend, put inside a packet of Whatman-41 filter paper, was immersed in 50 ml of methyl ethyl ketone (MEK) solvent at room temperature and the solvent was renewed every 24 h. After complete extraction as measured from constant residual weight, the samples were removed and dried to constant weight using a vacuum oven at 70 °C. The weight percent of entrapped FKM into polyamide matrix was calculated from the final and the initial weight of the rubber in the blends. Similar procedure for solubility in formic acid was followed to calculate the weight percent of unextracted polyamide.

2.4.2. IR spectroscopy in attenuated total reflectance (ATR) mode

IR spectra (in ATR mode) of the pristine polymers and the blends were taken on smooth film using a Perkin–Elmer (model 400) spectrophotometer with a resolution of 4 $\rm cm^{-1}$ and sixteen scans were averaged for each spectra.

2.4.3. Microscopy

2.4.3.1. Field emission scanning electron microscope. Morphology of various blends was recorded from FESEM (S4800 Hitachi microscope) at an acceleration voltage of 15.0 kV at a working distance of 6 mm. A thin layer of Platinum was sputter coated for 60 s on the smooth sample surface to avoid charging on exposure to electron beam during FESEM analysis. Measurement of particle diameters (d_i) from the photomicrographs gave the rubber

(a) PA6

(b) FKM

Fig. 1. Structure of (a) PA6 and (b) FKM.

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