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Thermal and mechanical characterization of injection moulded high density polyethylene/paraffin wax blends as phase change materials

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ABSTRACT

Thermal and mechanical properties of blends based on high density polyethylene and paraffin wax were investigated. The blends were prepared from 5 to 50 vol. % of paraffin wax employing a twin-screw extruder. Thermal behaviour of samples was determined by differential scanning calorimetry, thermogravimetric and dynamic mechanical analyses. A displacement of melting temperature of polyethylene was detected as a consequence of the plasticization effect of wax. These results revealed that melting temperatures and latent heats of samples are suitable for their application as phase change materials. Blends were processed by injection moulding which is an advantageous method to obtain pieces of this kind of materials. The evolution of loss tangent versus temperature of injected samples showed the lack of miscibility between the components of the blend. Tensile tests were carried out to characterize the mechanical strength of blends. Elongation at break decreased as paraffin wax content increased, and Young's modulus decreased with wax content but in the case of blends with a 30 vol. % of wax and more, brittle rupture occurred and no yield point was observed.

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1. Introduction

Phase change materials (PCMs) are substances with high heats of fusion that are able, through melting and solidifying at certain temperatures, to store and release large amounts of energy [1]. These materials store energy in the process of changing their aggregate state from solid to liquid. When PCMs reach the temperature at which the phase change takes place, large amounts of heat are absorbed without they get hotter. When the ambient temperature in the space around the PCM drops, the PCM solidifies, releasing its stored latent heat. PCMs absorb and emit heat while maintaining a nearly constant temperature [2]. These materials must have a large latent heat and high thermal conductivity. They should have a melting temperature lying in the practical range of operation, melt congruently with minimum subcooling and be chemically stable, low in cost, non-toxic and non-corrosive [3]. Materials that have been studied are hydrated salts, paraffin waxes, fatty acids and

eutectics of organics and non-organic compounds. PCMs provide solutions in very specific areas, for example related to the time delay and available power between production or availability of energy and its consumption in receiving systems (solar energy, cogeneration, etc) and in applications of thermal protection [4].

Commercial paraffin waxes are cheap with moderate thermal storage densities ($\sim 200 \text{ J/g}$), they undergo negligible subcooling, and they are chemically inert and stable with no phase segregation. However, they have low thermal conductivity ($\sim 0.2 \text{ W/m}^{\circ}\text{C}$) which limits their applications as passive systems in PCM [3]. The encapsulation for preventing leakage of melted PCM would decrease the thermal conductivity, so another possibility to keep waxes in a stable shape during their application is to blend them with convenient polymers. Blends of high density polyethylene (HDPE) and several kind of paraffin wax (PW) become suitable materials for applications as phase change materials [5–10]. Inaba et al. [5] obtained a shape-stabilized paraffin (pentacosane) as a solid-liquid phase change material employing HDPE as a supporting material. The test shape-stabilized paraffin was composed of the paraffin 74 mass% and HDPE 26 mass%. The whole compound can keep the same shape in a solid state without leakage of the melted paraffin even if the paraffin melts during a heat storage process. The work carried out by Hong et al. [7] deals with the preparation of HDPE and paraffin wax blends and the analysis of the structure by using





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scanning electron microscopy. On the other hand the thermal properties were studied. Sari [8] focused his work in the preparation of samples and the determination of their thermal properties. In order to improve the thermal conductivity, expanded and exfoliated graphite was added to the melted composite. Molefi et al. [9] presented the preparation of PCM based on paraffin wax and three different matrices as LDPE, LLDPE and HDPE. The samples were melt pressed and thermal properties were determined.

Paraffin wax is the phase change material and HDPE serves as the supporting material, which provides structural strength and prevents the leakage of the melted paraffin. In this thermal energy storage system, while dispersed paraffin wax changes in state from solid to liquid, the HDPE keeps the material in a compact shape. As long as the operating temperature is below the melting point of the supporting material, the compound material can keep its shape even when the PCM melts during a heat storage process. The net structure of the PCM prevents leakage of liquid to occur when paraffin melts. Lee et al. [6] studied the durability of injected high density polyethylene/paraffin blends as a thermal energy storage material by investigation of the seepage behaviour of paraffin. They also determined that the crystalline morphological characteristics of HDPE are responsible of the excellent sealant property of the blend. Mngomezulu et al. [10] characterized melt pressed blends of HDPE, a Ficher-Tropsch paraffin wax and alkali-treated wood flour, and concluded that wax influences the viscoelastic behaviour of the HDPE matrix in the blends.

Plastic injection moulding, which is a very cost effective method to produce very large quantities of parts, involves heating the material to a temperature at which flow is possible, forcing the plastic to pass through a nozzle into a shaped cavity, and cooling it. HDPE/PW blends are presented as phase change materials suitable for applications where it is necessary to maintain stable the temperature. The melting temperature of wax establishes the field of application, and dissipation of heat in several devices is a critical point where these materials would be able to play an important role. So, they can be employed as thermal protection systems of electronic devices.

In this investigation, injection moulding is proposed as a profitable method to process phase change materials, and provides advantages not only related to this kind of processing route but final mechanical properties of parts as well. The evaluation of thermal and mechanical properties of injected HDPE/PW samples was realized under the requirements of PCMs by means of DSC, TGA, DMA and tensile tests.

2. Materials and methods

The materials employed were a high density polyethylene supplied by Dow Plastics with a MFI of 25 g/10 min (190 °C and 2.16 kg), and a soft paraffin wax which is refined from petroleum, supplied by Panreac (carbon distribution C18–C50).

Pure components were firstly mechanically mixed in a Turbula for 15 min, and then they were blended in a twin-screw extruder Haake Rheomex CTW100p at 160 °C and 40 r.p.m. in order to obtain larger amounts of sample. Blends with different volume percentages of paraffin (5, 10, 20, 30, 40 and 50) were prepared with no leakage of this component.

Differential scanning calorimetry (DSC) experiments were carried out in a Perkin Elmer Diamond calorimeter with nitrogen as purge gas. Samples with a mass of ~10 mg were sealed in a 50 μ l aluminium pan, and an empty pan was used as reference. They were heated from 20 to 160 °C at a heating rate of 10 °C/min and then cooled at the same rate. Subsequently, they were heated again at the same conditions. Peak temperatures and enthalpies of melting were determined from the second scan.

Thermogravimetric analysis (TGA) was performed in a Perkin Elmer TGA1 thermogravimetric analyzer from 30 to 600 $^{\circ}$ C at a heating rate of 10 $^{\circ}$ C/min in nitrogen atmosphere.

Rheological experiments were carried out in a capillary rheometer Haake Rheocap S20. The dimensions of the die were 1 mm of diameter and a length of 30 mm in order to keep a L/D ratio of 30. The shear rate was chosen in a range from 100 to $10,000 \text{ s}^{-1}$ and a melting time of 5 min was employed for each test.

The parts were conformed in an Arburg 220S 250-60 injection moulding machine. The injection parameters were optimized: the temperature profile of the barrel was set to 145/150/155/160 °C from the feeding zone to the die the mould temperature was 40 °C. The holding pressure profile was established in three steps from injection pressure till 25 bar.

Dynamic mechanical analyses (DMA) were performed in single cantilever using a TA Instruments DMAQ800 instrument. A frequency of 1 Hz and an amplitude of 20 μ m were employed and samples were heated from -135 to 100 °C at a heating rate of 4 °C/ min. The width and thickness of the injected parts employed in DMA were close to 5.90 and 3.00 mm. The maxima on tan δ -temperature plot were determined to identify the relaxations associated to glass transitions.

Finally, mechanical properties of injected samples were determined through tensile tests in a Schimadzu AG-I testing machine at room temperature. The mechanical tests were carried out on the basis of standard EN ISO 527-1:1996, and the dimensions of the samples correspond to the specimen numbered as 5B. The deformation speed was 50 mm/min and results were obtained testing five parts of each blend.

3. Results and discussion

3.1. Determination of density

Pycnometric density of blends against wax content is plotted in Fig. 1. The dashed line represents density values calculated from rule of mixing [11]. When increasing wax content density of blends decreases. Experimental results agree with theoretical ones which indicates that there was no paraffin wax loss during mixing.

3.2. Differential scanning calorimetry

Fig. 2 shows DSC heating scans of pure components and blends. In the case of pure HDPE there is only one peak at 130.7 $^{\circ}$ C and, in the case of pure PW, two endothermic peaks at 32.3 and 53.1 $^{\circ}$ C are



Fig. 1. Pycnometric density of HDPE/PW blends.

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