



## Research paper

## Structure and properties of clay/recycled plastic composites

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## ABSTRACT

We are presenting a clay (montmorillonite) based method of reintroducing plastics back into the market without subjecting them to extended processing methods. We have prepared montmorillonite/recycled polymer materials with recycled polystyrene (R-PS) and recycled polyethylene (R-PE). R-PS was melt mixed with as-received organomodified montmorillonite or blowing agent treated organomodified montmorillonite which led to intercalated/exfoliated clay/polymer nanocomposites. Similarly, R-PE was melt compounded, with or without the addition of a compatibiliser with the above mentioned organomodified clay minerals which resulted in conventional composite formation. In the case of R-PS, the thermal degradation temperature of the materials increased with the presence of clay minerals, whereas for R-PE based materials it was observed that the thermal degradation temperatures decreased with the presence of clay minerals. Overall it was observed that the presence of clay minerals improved the stiffness of the materials. The use of blowing agent treated organomodified clay minerals in R-PS led to nearly doubled impact strength compared to organomodified clay/R-PS nanocomposites.

## 1. Introduction

During the life cycle of a plastic material and depending on the environment in which the material is used, the polymer may undergo thermo- and/or photo-oxidative degradation, leading to irreversible changes at molecular and morphological levels (Kartalis et al., 2001; Pospíšil et al., 1995). These changes to the structure of the polymer are typically more pronounced when material recovery is performed. Mechanical recycling is an energy effective plastics recovering process that uses mechanical processes (e.g. separation, washing, shredding and processing) to recover polymeric materials from the recycled plastic stock (Finnveden et al., 2005; Vilaplana and Karlsson, 2008). However, the mechanically recycled polymers are typically characterised by inferior mechanical properties, compared to the pristine materials (Kartalis et al., 2001), which may be due to thermo-mechanical deterioration that may occur during the recovery process (Strömberg and Karlsson, 2009; Vilaplana and Karlsson, 2008).

Thermo-oxidative and thermo-mechanical degradation of polymer chains and the possible presence of unwanted degraded chemical substances make interesting the use of additives that are able to minimise the impact of these undesirable products. Over the years, a myriad of materials (such as: stabilisers, compatibilisers and particles) have been used in order to diminish the impact of thermo-oxidative and thermo-

mechanical degradation experienced by the plastic materials (Fortelný et al., 2004; Vilaplana and Karlsson, 2008). The well-known ability of clay minerals to adsorb and absorb chemical substances and the beneficial improvement of thermal, mechanical and barrier properties with the dispersion of small amounts of clay minerals in pristine polymers and polymer blends make clay an ideal candidate to aid in the recovery of plastic materials (Chaiko and Leyva, 2005; Katti et al., 2006; Lee et al., 1997; Liu et al., 2000; Okada and Usuki, 2007; Zhao et al., 2005).

Clay minerals are ubiquitous in nature, have the ability to absorb harmful substances that might be present in the recycled stock and each clay layer is characterised by superior strength and stiffness compared to any polymer matrix (Chen and Evans, 2006). The effects of adding natural bentonite (i.e., sodium montmorillonite, Cloisite® Na) or organomodified bentonite (i.e., Cloisite® 25A) in recycled polyethylene terephthalate (PET) have been structurally and mechanically evaluated for different clay mineral loads (Pegoretti et al., 2004). It was observed that the dispersion of organomodified bentonite resulted in intercalated clay nanostructures, whilst natural bentonite presented mostly as aggregates. The tensile properties showed that the modulus increased with clay minerals load augmentation and the tensile strength climaxed at 5 wt% clay regardless of the type of clay mineral used (Pegoretti et al., 2004). The formation of intercalated and exfoliated nanostructures increases the exposure of the surface of the clay layers and

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allows for the stress to which polymer matrix is subjected to transfer to the nanostructure so as to withhold superior loads. The effect of 5 wt% organomodified bentonite dispersion into another recycled polyester, i.e., poly(butylene terephthalate) (PBT) has also been investigated (Quispe et al., 2015). It was observed that the type of organic modifier influences the morphology of the polymer nanocomposite. Partially exfoliated clay/polymer nanocomposites were obtained when using single tail tallow (i.e., Cloisite® 25A) and only intercalated nanostructures occurred when a double tail tallow (i.e., Cloisite® 20A) was used. The partially exfoliated nanocomposites presented a better dispersion of the nanofiller and the higher improvements in the tensile modulus and the tensile strength over recycled PBT when compared to intercalated Cloisite® 25A/PBT nanocomposites (Quispe et al., 2015). The dispersion of organomodified bentonite (i.e., Cloisite® 30B) into recycled polypropylene with 30 wt% maleated polypropylene led to the formation of well dispersed composite materials characterised by highly intercalated nanostructures (Phuong et al., 2008). The mechanical properties showed progressive improvements with smectite augmentation with the highest values for tensile strength and Charpy impact strength being encountered for a clay load of 4 wt% (Phuong et al., 2008).

Analysing the average waste consumption of a household, it was discovered that thermoplastic waste represented 12% of the yearly household residue; from which polyethylene (PE) made up 75% and polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC) and PET represented 10, 8, 4 and 3%, respectively (Finnveden et al., 2005). Thus, the current study focuses on two major household waste thermoplastics, a non-polar polymer, i.e., PE, and a low-polar polymer, i.e., PS. This work examines the structure and thermal and mechanical properties of clay/recycled polymer composites manufactured with an as-received organomodified montmorillonite (Organoclay Nanomer® I.44P) and a blowing agent-treated organomodified montmorillonite. These treated clay minerals have been previously used to manufacture clay/polymer nanocomposites with a higher degree of exfoliation and superior properties (Istrate and Chen, 2014). It is hypothesised that by dispersing these clay minerals in recycled polymer matrices polymer composites/nanocomposites with better clay dispersion and superior properties will form. If this is the case, we hope that by using this procedure higher amounts of plastics will be recycled and reintroduced to the market and that the versatility of the products manufactured with recycled polymers will increase.

## 2. Experimental section

### 2.1. Materials

Recycled high-density polyethylene (R-PE) from Monnad Industries (Ireland), obtained from pelletizing milk jugs, was generously provided by Athlone Institute of Technology (Ireland). Recycled impact-modified polystyrene (Axpoly® PS01), denoted from here on as R-PS and representing 100% post-consumer recycled polymer recovered from refrigerators, was generously supplied by Axion Polymers (UK). R-PE and R-PS were used as polymer matrices for the manufacturing of clay/polymer composites. For R-PE a compatibilising agent, i.e., polyethylene-grafted-maleic anhydride (PEgMA) was used. PEgMA was purchased from Sigma-Aldrich Ireland Ltd. (Ireland). Organomodified montmorillonite Nanomer® I.44P (Clay), a dimethyl dihydrogenated tallow ammonium chloride (2M2HTA) modified montmorillonite, manufactured by Nanocor Corporation (USA), was kindly supplied by Nordmann, Rassmann GmbH (Germany). The organic content of the organomodified montmorillonite was previously determined from loss on ignition test to be 40% (Istrate et al., 2012). The as-received organomodified montmorillonite was treated with azodicarboxamide (ADC), a well-known blowing agent, following a procedure described in our previous publication (Istrate and Chen, 2014). The resulting clays were denoted as ADC-Clay.

### 2.2. Nanocomposite manufacturing

R-PS, R-PE and compatibilised R-PE (R-PE/PEgMA = 90/10, w/w) with 4 wt% clay layers were manufactured on a Prism twin screw extruder (UK) with 16 mm-diameter screws and a length to diameter ratio of 25. The materials were passed three times through the twin-screw, once at a screw speed of 200 rpm and then twice at a screw speed of 100 rpm. For the organomodified montmorillonite nanocomposites the temperatures were maintained at 160, 170, 175, and 180 °C from hopper to die, for all three processes. For the blowing agent-treated organomodified montmorillonite the temperatures were maintained at 160, 170, 175, and 180 °C when the material was processed at 200 rpm and increased to 165, 175, 190, and 200 °C when the material was processed at 100 rpm. After passing the material through the extruder, the extrudates were water cooled and pelletized. Tensile and impact specimens were manufactured on a bench top injection moulder (Ray Ran model 2 Test Sample Injection Moulding Apparatus, UK). The injection moulder was used at a barrel temperature of 220 °C, a tool temperature of 55 °C and a pressure of 0.76 MPa for R-PE materials and a barrel temperature of 210 °C, a tool temperature of 55 °C and a pressure of 0.76 MPa for R-PS materials.

### 2.3. Characterization

X-ray diffraction (XRD) was carried out on a Phillips PW1720 X-Ray Diffractometer with a  $\text{CuK}\alpha_1$  ( $\lambda = 0.15406$  nm) anode tube at standard conditions of 40 kV and 20 mA. The samples were tested from 2° to 10°, 2 $\theta$  angle, at a step size of 0.02° and a duration of 2.5 s per step. Powder samples were used for the clay minerals, while thin samples (1 mm thick) were used for the composite materials. These samples were prepared by applying a pressure of 5.1 MPa for 10 s at 210 °C.

Transmission electron microscopy (TEM) was performed on a TECNAI G2 20 Twin electron microscope at 200 kV accelerating voltage. The specimens were ultramicrotomed using a Reichert-Jug ‘Ultracut’ equipped with a diamond knife. The sections (~100 nm in thickness) were collected in a trough filled with water and then placed on a 200 mesh copper grid.

Scanning electron microscopy (SEM) imaging on tensile fractured surfaces was performed using a Zeiss Ultra Scanning Electron Microscope (for R-PE materials) or on a Tescan Mira Variable Pressure Field Emission Scanning Electron Microscope (for R-PS materials) at a voltage of 5.0 kV. Prior to being analysed the samples were mounted on stubs and their surface were platinum or gold coated.

Thermogravimetric analysis (TGA) was performed on a Perkin Elmer Pyrus 1 TGA equipped with an ultra-micro balance with a sensitivity of 0.1  $\mu\text{g}$ , under nitrogen flow (20 mL·min<sup>-1</sup>), from 100 °C to 650 °C at a heating rate of 10 °C·min<sup>-1</sup>.

The tensile tests were run according to ISO 527:1996 on a Zwick Z005 machine (Germany). Five dog bone specimens (Type 1BA) were tensile tested using a 5 kN load cell and a testing speed of 20 mm·min<sup>-1</sup> for R-PE materials and a 2.5 kN load cell and a testing speed of 5 mm·min<sup>-1</sup> for R-PS materials. Impact tests were run according to standard ISO 179:1997 at room temperature on a Charpy impact tester (JinJian XJJD-5, China). The tests were run at a speed of 2.9 m·s<sup>-1</sup> and using a hammer of 2 J for R-PE materials and 0.5 J for R-PS materials. Seven specimens (80 mm × 10 mm × 4 mm, length × width × thickness) were impact tested for each batch of materials. Prior to being tested the impact specimens were notched with a type A notch, using a 45° cutter and a milling machine. The mean and standard deviation values reported for the mechanical tests represent a confidence level of 95%. Statistical significance was assessed by a Two-tailed, Type II ‘t’ test with a criterion that the probability of a difference in means due to chance is smaller than 0.05.

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