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Novel silicon microparticles to improve sunlight stability of raw polypropylene



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ABSTRACT

Oxidation of polyolefins by ultraviolet/visible irradiation is a significant limitation for their use in several technological applications. The use of high-tech additives such as silicon microparticles becomes a compositing strategy that can improve the performance of these materials at long-term service conditions. Silicon particles were added to non-additivated raw polypropylene (PP) prepared by hot melt extrusion and subjected to accelerated sunlight irradiation tests. The stability of thermal properties, mechanical performance and thermal decomposition behaviour of composites was evaluated by differential scanning calorimetry, dynamic mechanical–thermal analysis and thermogravimetry. This paper shows the potential of silicon microparticles to protect PP from degradation during both processing and accelerated sunlight irradiation. The addition of polydisperse silicon microparticles to the raw polypropylene preserved the appearance and improved the thermal behaviour, mechanical properties and thermal stability. It was found that silicon particles at 5 wt% provided a suitable protection of raw non-additivated PP against sunlight irradiation.

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1. Introduction

Polypropylene (PP) is one of the most extensively used polymers in several industrial sectors due to its attractive properties such as suitable processability, feasible mechanical properties, low price and low weight, among others [1]. Wiring, construction, automotive and packaging are some of the main areas. Therefore, the stabilization against different factors which cause polymer degradation such as UV-radiation, temperature, stress loading, water absorption and weathering, among others, is considered crucial for long-term applications.

It is well known that the main effects of photo-oxidation of polypropylene consist in the reduction in molecular size, crystallisation and formation of extra chemical groups, as hydroperoxides and carbonyl species such as ketones, esters and acids [2–6]. Several protective measures have been thus proposed to increase stability against sunlight irradiation and different kinds of additives have been considered.

Hindered amine light stabilizers (HALS) were introduced more than 25 years ago and they led to considerable improvement of polyolefin light stability based on radical scavenging in which nitroxides play a key role reacting with alkyl radicals and forming amino ether, but some drawbacks have been also observed [7–9]. The basic nature of HALS causes that some

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acidic substances have a large negative effect on its effectivity. Thus, high temperatures along with HCl, HBr or HNO₃ and, in general, acid rain which are present in the atmosphere of developed countries due to pollution may deactivate the protective capability of HALS [10].

UV-absorbers used solely or in combination with other light stabilizers are considered other group of additives to prevent photo-degradation. Combinations with nickel derivatives were used, especially for the stabilization of polyethylene films applied in agriculture and horticulture [11]. These substances absorb the harmful UV light more effectively than the polymer and can transform the excess of energy as heat or less-harmful radiation. A main disadvantage of UV absorbers, however, is the necessity of certain sample thickness that permits a proper absorption depth [9,12,13]. Moreover, as well as described below for HALS, UV absorbers possess an intrinsic durability which can turn in a considerable drawback. Sun irradiation near-IR reflective pigments, based on light scattering like titanium dioxide, zinc oxide or light absorption as carbon black have been considered to prevent polymers from photo-degradation [14–18]. Nevertheless, the majority of these systems consist in the addition of nanoparticles, which can provoke toxicity and harmful effects when inhaled or ingested by living organisms [19]. In this line, a novel micro-sized material has been recently developed which can improve significantly resistance to photo-degradation when added to a polymeric matrix in comparison with conventional screeners, preventing from harmful drawbacks. Silicon nano and microparticles obtained either by chemical methods [20–22] or through milling process [23], block very efficiently sun radiation in the whole UV, visible and infrared regions when coated on a surface. A 20 μm thick coating of silicon microparticles is able to block 99% of the whole sun radiation. This protective mechanism is due to huge value of the refractive index much larger than that for any other material, as titanium dioxide, used for light protection [20].

The aim of the present study concerns determining the capability of the polydisperse silicon microparticles for sunlight protection when added to a non-additivated raw polypropylene matrix. In order to avoid problems derived of phase separation between matrix and particles, some additives acting as coupling agents can be added to the composite. Maleic Anhydride (MAH) grafted PP has been proved to be an effective functional molecule for the reactive coupling between polypropylene (PP) and other polymers [24,25] and loadings [26–31]. Concretely, for silicon microparticles the use of MAH prevented the composites from the phase separation and conglomeration of silicon domains. Moreover, the inclusion of micron-sized particles usually affects polymer properties and behaviour, as crystallisation process, melt processing, thermal stability or mechanical properties, which have to be studied [32–35]. Therefore, a composite of grafted MAH–PP blended with non-additivated raw PP and silicon microparticles was considered for the study. For this purpose, an initial characterisation of the materials prior to the exposition should be accurately considered. Then, in order to determine how these particles avoid the degradation process, a monitored accelerated sunlight irradiation procedure was carried out.

2. Experimental procedure

2.1. Materials description and sample preparation

Polydisperse silicon microparticles were obtained from metallurgical grade silicon powder (Silgrain[®] with 99.5% of purity from Elkem), through a process of grinding. Screening of the particles was performed achieving that the 98% were smaller than 2 μm (see Fig. 1) [23].

Unstabilised isotactic PP ISPLEN PP040 G1E (MFI 3 g/10 min at 230 °C) was supplied by Repsol YPF (Spain) as pellets. Maleic anhydride grafted polypropylene (MAH–PP) from Eastman (Tennessee, USA), coded as G-3003 with a maleic anhydride content of 1%, a molecular weight of 52,000 g/mol and a viscosity of 60,000 cP (measured at 190 °C) was used to ensure coupling of the polypropylene matrix with Si microparticles.

PP, MAH–PP and Si were mixed considering different compositions and resulting specimens were labelled as follows: PP (raw polypropylene), PP–MAH (PP mixed with a 3 wt% of MAH–PP), and PP–MAH–3Si and PP–MAH–5Si (PP–MAH mixed with 3 wt% and 5 wt% of Si, respectively). Mixtures were prepared in a laboratory-scale co-rotating extruder working at 40 rpm and a temperature program of 185–190–195 °C from feeder to die. Afterwards, 200 μm thick probes were obtained by hot compression moulding at 190 °C, following a force (kN) time (min) program of (i) 0–20, (ii) 440–3, (iii) 660–3 and (iv) 880–4, followed by a quenching step to room temperature through water immersion.

2.2. Accelerated sunlight irradiation procedure

Accelerated sunlight irradiation was carried out by exposing the samples to controlled cycles of intense light into an Atlas Suntest XLS + Xenon Exposure equipment (Illinois, USA) with black panel temperature of 50 °C and irradiance of 600 J s⁻¹ m⁻² in the UV–visible range wavelength ($\lambda = 280\text{--}800$ nm). Several irradiation stages were considered at 250, 750, 1500, 2250 and 3000 h. Even though such testing cannot reproduce all the elements of weathering and exactly predict the performance of these samples in a real outdoor exposure, some meaningful relative comparisons can be done. As a reference, the data at Jacksonville, Florida (USA), with an annual total radiant energy of 5800 MJ m⁻² ($\lambda = 280\text{--}3000$ nm) are considered. According to bibliography [36], this total radiation in the UV–visible range ($\lambda = 280\text{--}800$ nm) would correspond to 3248 MJ m⁻². Thus, the equivalence of 1 h of simulated irradiation would be equivalent to 5.83 h of real exposure in Jacksonville, Florida. Table 1 shows the equivalence for the different stages of irradiation.

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