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Material properties

Effect of residual contaminants and of different types of extrusion processes on the rheological properties of the post-consumer polypropylene

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ABSTRACT

Rheological measurements were conducted to verify the influence of different mechanical recycling processes and the presence of contaminants on the degradation of post-consumer polypropylene. Firstly, polypropylene (PP) was contaminated to simulate a post-consumer material, following the protocol recommended by the FDA. PP was subsequently recovered (washed and dried) and the samples were submitted to different extrusion processes. The rheological data demonstrated that the different types of processing applied and the presence of contaminants altered the molecular structure of the samples. The contaminants acted as agents that accelerated the polymer degradation. The contaminated samples submitted to higher shear rates exhibited greater decrease in their molar mass and a slight narrowing in the molar mass distribution. Also, it was observed that the most degraded samples showed decrease in their molar mass, in the viscosity and in the level of their molecular entanglements. These samples also exhibited a more Newtonian behavior and their molar mass distribution showed a slight narrowing. By calculating the ratio of the molar mass it was possible to quantify the degree of degradation of PP samples, confirming the results obtained.

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1. Introduction

One of the most commonly used polymers in food packaging is polypropylene (PP), due to its low cost and good properties, mainly the mechanical ones. Because of its wide utilization in disposable products such as food packaging, waste PP has become a global problem. The low degradability of PP and the large volume that it occupies generate serious environmental problems when it is disposed of in landfills and dumps.

A sustainable and attractive solution for this problem would be to recycle and reuse it for the same purpose, as food packaging. However, there are restrictions established by regulatory agencies [1–8] for the reuse of recycled polymers for direct contact with food. These restrictions were created due to the possible migration of contaminants from the recycled material into the food, which would endanger the health of consumers [9–13]. The U.S. Food and Drug Administration (FDA), which is the regulatory agency in the United States, published one of the most important documents in the area called the *challenge test*. This document was developed to evaluate the efficiency of the recycling processes in decontaminating the post-consumer polymer [3]. For this, the virgin polymer is exposed to

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selected surrogate contaminants and then it goes through the recycling process. Afterwards, the final material is analyzed and the contaminants that migrated to the different food simulants can be identified and quantified.

Regarding this issue, most studies that considered the feasibility of recycled materials for contact with food have focused on the migration of contaminants [9–13]. However, it is also important to know whether the polymer can retain its properties after the recycling process in order to make its reuse possible.

Mechanical recycling, although widely used, can lead to degradation and modification of the properties of the PP. The combined action of physical factors (heat, light, radiation and shear) and chemical factors (oxygen, catalytic residues and additives), as well as the influence of the presence of glues, paints, adhesives and other impurities, can degrade the polymer through chain rupture [14–22]. This degradation occurs mainly because the β-scission of tertiary carbons generate double bonds and free radicals that react with oxygen to form peroxides and hydroperoxides, which produce more radicals, making the process autocatalytic [23,24].

Several studies have reported that changes in the molecular structure of the PP due to thermo-mechanical degradation can occur during its reprocessing [15,20,21,25–29]. Costa et al. [28] verified that during multiple cycles of recycling in a single-screw extruder the PP was degraded by scission chain. The authors observed that the degradation increased in each cycle, consequently decreasing the molar mass of the polymer.

Some studies [30–32] have shown that the action of degrading agents on the rheological behavior of the polymer can occur. Azizi and Ghasemi [32] examined the reactive extrusion of PP with various concentrations of dicumil peroxide (DCP) in a co-rotating twin-screw extruder. It was noticed that the higher the concentration of the initiator (DCP) in the polymer, the lower the viscosity, leading to higher flow rate.

Regarding the presence of impurities, Incarnato et al. [33] evaluated the effect of contaminants such as phenylcyclohexane, benzophenone and di-n-butyl phthalate on the degradation of the PP submitted to several cycles of injection molding. Gel permeation chromatography (GPC) measurements have shown that the molar mass decreased in the presence of these contaminants, more significantly after the second cycle.

In a previous paper, we analyzed the influence of different types of mechanical recycling processes to decontaminate PP samples by removing volatile and non-volatile contaminants (benzophenone, chloroform, tetracosane and toluene). The results showed there was a significant influence of the type of the processing adopted to decontaminate the samples [34]. In the present study, the effect of the presence of contaminants and of the different processing adopted were evaluated, observing the molecular changes that occurred during the recycling of the post-consumer PP. Small amplitude oscillatory shear (SAOS) tests were performed in order to study the rheological behavior of the samples. The main objective is to understand more about the structural changes that occurred in the recycled polypropylene.

2. Materials and methods

2.1. Materials

The study was conducted using a commercial homopolymer polypropylene (HP502H) supplied by Braskem Company, Brazil. The MFI was 2.2 g/10 min (230 °C, 2.16 N – ASTM D-1238), and the density was 0.905 g/cm^3 (ASTM D-792), according to the supplier. The contaminants used were benzophenone (Acros Organics – 99%), chloroform (Synth – 99.8%), tetracosane (Alfa Aesar – 99%) and toluene (Chemis – 99.5%) dissolved in 2-propanol (Vetec – 99.5%) and heptane (Vetec – 99.5%).

2.2. Sample preparation

2.2.1. Contamination process

PP pellets were contaminated using a cocktail of contaminants according to the FDA guidelines [3], thereby simulating a post-consumer polymer. These contaminants represent a wide variety of potential chemicals that may contaminate consumers, and the concentration of these compounds was extremely high to represent the worst-case scenario. The surrogate cocktail was comprised of chloroform, toluene, tetracosane and benzophenone [3]. The chloroform and toluene represent components of cleaning solvents, benzophenone simulates non-volatile polar pesticides and tetracosane simulates motor oil.

In the contamination process, the virgin pellets were placed inside a hermetically sealed container and immersed in a solution containing the surrogate contaminants for 14 days at 40 °C with periodic agitation [3,34].

2.2.2. Recovery process

After the contamination process, the samples were recovered (washed and dried) in a MA037 oven with air renewal [35]. In this process, the materials were prewashed with water for 10 min ($T_{water} = 24$ °C) and, subsequently, washed in a solution of 1% NaOH for 5 min and rinsed with water for 10 min. After the washing, the PP samples were dried for 24 h at 50 °C.

2.2.3. Reprocessing of the samples

To test the effect of each extrusion process on the degradation of the PP samples and the effect of the residual contaminants on their molecular structure during the processing, three types of extrusion process were used. The samples were processed as follows:

- In a single-screw extruder (Gerst) (D = 25 mm, L/D = 24) with a temperature profile of 180, 180 and 200 °C and screw rotation speed of 80 rpm;
- In a co-rotating twin-screw extruder with forced degassing (Werner & Pfleiderer) (D = 18 mm, L/D = 40) under a vacuum of 0.6 bar. The temperature profile was 210, 230 and 232 °C and screw rotation speed was 700 rpm. The vacuum degassing system was chosen because it removes gases during the process;
- In a cascade extruder (Gerst) (D = 25 mm, L/D = 24) with a temperature profile of 180, 180 and 200 °C and

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