

Recycled Poly Chain Extension by a Reactive Extrusion Process

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INTRODUCTION

Post-consumer PET undergoes a reduction in intrinsic viscosity, $[\eta]$, when recycled in a normal extrusion system. The occurrence of thermal and hydrolytic degradation reactions during recycled PET melt processing is responsible for the reduction in $[\eta]$ or molar mass of the PET. The presence of water and polyvinyl chloride (PVC) in the recycled PET flakes produces PET chain scission during normal extrusion. At processing temperature (280[degrees]C), hydrolysis reactions occur between water and PET, resulting in shorter chains with carboxyl and hydroxyl end groups. The thermal cleavage of the PET ester bond results in PET chains with carboxyl and vinyl ester end groups. Recently, intensive drying to remove moisture, and vacuum degassing processing, were introduced by Erema in their plastic-recycling systems to minimize the effect of these reactions, resulting in higher PET $[\eta]$ in comparison with normal extruded PET (1). Solid state processing has also been reported to achieve higher PET $[\eta]$ (2, 3). However, this process is considered to be slow and expensive. In this study, chain extension by reactive extrusion was chosen to overcome the reduction of $[\eta]$ for several reasons. It is less expensive than solid state processing and easier to apply in an existing normal extrusion system, and because of the proven success of the chain extension process with virgin and recycled PET (3).

In order to raise the $[\eta]$ of the PET to a higher desired level, reactive blending of virgin PET with chain extenders was investigated by Inata and Matsumura (4-9). Their efforts concentrated on evaluating potential chain extenders. They showed that additive type di- or poly- functional chain extenders were the preferred chain extenders because of their high reaction rate without generating by-products.

PMDA has previously been reported as an efficient chain extender or branching agent (10, 11). It is thermally stable, produces no side products on reaction with PET, and is tetra- functional, commercially available, and economical. Khemani (10) showed that when 0.2 to 0.3 wt% PMDA was used with virgin PET, a significant increase in melt strength was obtained. Because of these results, PMDA was the selected chain extender in this study; its concentration was chosen to be one of the process variables. The influence of variables such as the chain extender concentration and the reaction time on the virgin and recycled PET chain extension process has been investigated (4-17).

Chain extender concentration has been the most important parameter examined in reactive blending process research in recent years (14, 15, 18, 19). The theoretical amount of chain extender required was calculated according to the chain extension reaction stoichiometry. Chain extender in amounts higher than theoretically needed leads to crosslinking reactions, resulting in gel formation.

Akkapeddi (20) investigated a reactive extrusion system of virgin PET with added chain extenders. He obtained lower carboxyl content using different types of chain extenders.

Different models have been introduced to describe the reactive extrusion system. Janssen (21) introduced an interaction chart that presented the parameters affecting reactive extrusion. These parameters and conditions influencing the reactive extrusion process and its stability have been described by many researchers (21-24). Vergnes et al. (25) developed a model and software to characterize the flow in a co-rotating twin-screw extruder. Thermal, hydrodynamic and chemical reactions were found to be the main cause of instability in the reactive extrusion process. Controlling the causes of these instabilities and then minimizing them is the key to successful reactive extrusion.

It is well known that in general, an increase in reaction conversion can be expected as a result of an increase in residence time (22). In this study, the residence time was considered as the process variable, and its effect on the process stability and product properties was examined.

So far, there have been no reported studies describing a recycled PET reactive extrusion system implemented on a commercial-size extruder. This study investigates the reactive extrusion of recycled PET with a PMDA chain extender, in order to produce reactive extruded recycled PET material with high intrinsic viscosity.

EXPERIMENTAL PROCEDURE

Reactive extrusion experiments were performed using a commercial PET co-rotating twin-screw extruder at Visy Plastics. Melbourne facilities. Table 1 shows the normal extrusion operating conditions. PMDA was obtained from Nippon Shokubai Co. Ltd. with a purity of 99.7%, and recycled PET having an $[\eta]$ of 0.75 dl/g was obtained from Visy Plastics. Flakes of recycled PET were subjected to intensive drying at 170[degrees]C before being fed to the extruder. The flakes were dried by blowing desiccated air (dew point < -20[degrees]C) at 170[degrees]C through them for 4 hours. The average moisture content after drying was about 500 ppm. The reason for the relatively high moisture level of the recycled flakes is the limited drying time. Less moisture content could be achieved using a drying time longer than 4 hours; however, the commercial PET recycling industry limits the drying time of the flakes for economic reasons. The relatively high moisture content in the flakes is compensated by the efficient vacuum system attached to the extruder. The PET (flakes) used in the experiments had an average content of 400 ppm of PVC.

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