New approach for rapid process stabilization of plastics

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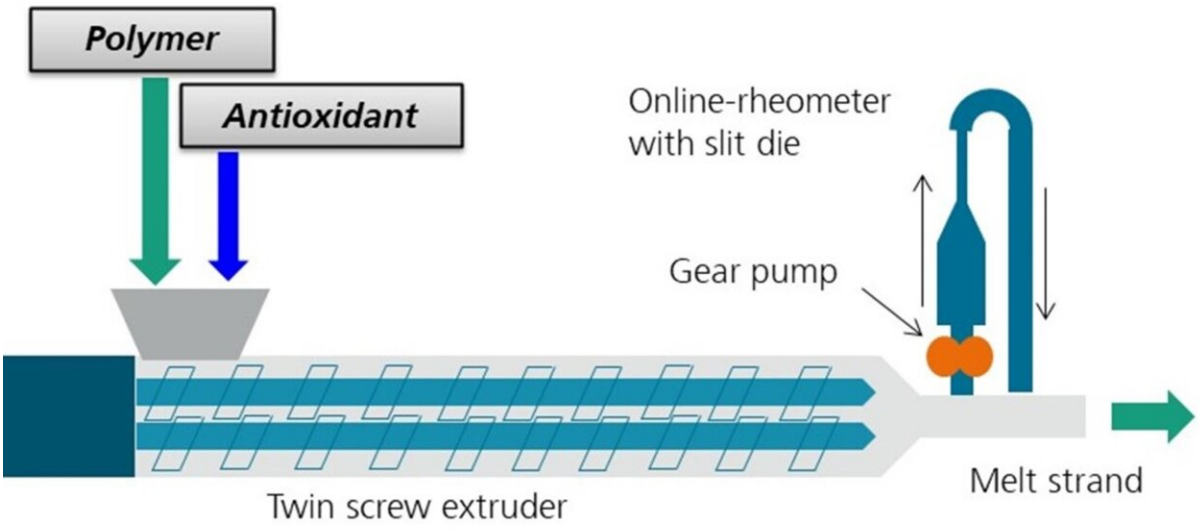

Scheme of the experimental setup with twin screw extruder and online rheometer. Credit: Fraunhofer LBF

Plastic materials are prone to degradation by atmospheric oxygen. These auto-oxidation reactions occur at ambient temperature but become especially relevant during melt processing. Adding antioxidants to the polymers results in a pronounced slowing-down of the oxidation processes. Only in this way, the production of plastic parts by injection molding, for example, is possible.

Developing new plastic grades is accompanied by lengthy experimental

procedures to optimize the antioxidant content. For Fraunhofer Institute for Structural Durability and System Reliability, these online rheological investigations are a promising method for accelerating the development process.

Antioxidants

Organic matter, and therefore also plastics, degrade by auto-oxidation when in contact with air. This degradation is initiated by elevated temperature or light and propagates as a radical chain reaction that causes cleavage of the polymer chains. The latter are primarily attacked by the OH radical resulting in the formation of hydroperoxide moieties. These triggers follow-up reactions leading to regeneration of the OH-radical.

For optimum protection of the polymer, two different types of antioxidants must be added. The primary antioxidant, often containing a phenolic structure, quenches the OH-radical. Secondary antioxidants consist of sterically hindered alkyl-derivatives of functional groups, such as phosphites or thioethers. These react with the hydroperoxide without OH formation. Both types of antioxidant therefore act in a synergistic way. A typical commercially available stabilizer package containing both antioxidants in equal amounts was used in the described experiments.

Studying process stabilization

Commercially available virgin plastic grades are typically equipped with appropriate stabilizer packages ready for use. For the sake of resource efficiency and economy, the optimum content of the process stabilizer must be determined during the development of new plastic grades. Processing of used plastics to recyclates faces the same problem because the stabilizers have been regularly depleted during the previous life

cycle.

Compounding the mill charge to recyclates to be used for example in injection molding, requires adding stabilizers adjusted to the respective type of plastics and its stage of aging. The traditional way to optimize the stabilizer content is based on compounding a series containing varying amounts of antioxidants. The compounds are then characterized offline by means of different tests, such as the melt volume rate (MVR, DIN 1133-1) or the oxidative induction time (OIT, ASTM D3895-19). First reliable results are obtained only after the compounding step.

Online characterization offers new potential

Researchers at Fraunhofer LBF are working to obtain an indication regarding the efficacy of the actual stabilizer content during the compounding step. To this end, the viscosity of the melt is used as a recorded response while varying the recipe. This is realized by incorporating an online rheometer behind the screw tips of a twin screw extruder to measure the flow curves of the shear as well as the elongational viscosity.

The first experiments were carried out on a minimally stabilized virgin polypropylene (PP).

The amount of stabilizer added was varied at selected screw speeds. The reduced process-related degradation is immediately reflected in an increase in viscosity in the flow curves. Above a certain additive level there is no further increase in viscosity. This means that for the actual processing conditions, the stabilizer concentration has reached the limit above which no further improvement can be achieved.

Thus, online rheology provides the formulation developer with valuable information regarding the efficacy of a processing stabilizer during

compounding.

Furthermore, the flow curves of the different polymers are not identical. The information content of a flow curve is therefore much higher than that of a single numerical value from an MVR measurement. In addition, the flow curves of the elongational viscosity can be included in the evaluation. Supported by an appropriate AI-based system, online rheology appears to be a very promising tool in implementing stabilization during the production of recyclates with the ability of real-time adjustment to the aging stage of the mill charges.

Provided by Fraunhofer-Gesellschaft

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