EuroSciCon

3rd Edition of International Conference and Exhibition on

Polymer Chemistry

March 26-28, 2018
Vienna, Austria

Laurent Verny et al., Polym Sci, Volume 4
DOI: 10.4172/2471-9935-C1-008

POLYIMIDE SYNTHESIS VIA REACTIVE EXTRUSION

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The most common way to synthetize polyimides involves the polycondensation of diamine and dianhydride monomers in solution. The reaction is in general achieved in two steps; first, an intermediate poly(amic acid) is formed, which is further cyclized into polyimide upon appropriate heating. In this solution polymerization process, high boiling temperature solvents are mainly used, such as N-methyl-2-pyrolidone. As such solvents are hazardous chemicals, there is a real interest to develop an approach preventing their use. To this end, some research works have been recently reported concerning the imidization into an extruder of a poly(amic acid) solution in dichlorobenzene. The drawbacks of this process remain in the solvent elimination and the total reaction time (several hours). In the frame of our project, we developed a method for obtaining polyimides directly in solvent-free conditions via a reactive extrusion process, within typical extrusion residence times (5–10 minutes). Trial runs have been performed on 15 mL micro-compounder. The optimization of the shear conditions and of the reaction temperature allowed obtaining polyimides within a reaction time ranging from 3–5 minutes. The scaling up of this process has been carried out using a twin screw extruder (L/D = 60). The polyimides obtained were characterized by both ^{1}H and ^{13}C NMR. The molar masses of the polyimides were obtained by size-exclusion chromatography. FT-IR spectroscopy has been used to confirm the imide structure and the imidization conversion rate has been determined by DSC.

Recent Publications:

1. Sroog, C.E. *et al.* (1965) Aromatic polypyromellitimides from aromatic polyamic acids. *J. Polym. Sci. A Gen. Pap.* (3): 1373.
2. Silvi, N. *et al.* (2003) Method for preparing polyimide and polyimide prepared thereby. *US patent no.7053168*
3. Yu, H-C. *et al.* (2016) Kinetic Study of Low-Temperature Imidization of Poly(amic acid)s and Preparation of Colorless, Transparent Polyimide Films. *J. Polym. Sci. Part A: Polym. Chem.* 54 (11):1593
4. Schab-Balcerzak, E. *et al.* (2010) Influence of azobenzene units on imidization kinetic of novel poly(ester amic acid)s and polymers properties before and after cyclodehydration. *J. Appl. Polym. Sci.* 118 (5):2624
5. Liaw, D-J. *et al.* (2012) Advanced polyimide materials. *Progress in Polymer Science* 37 (7):907.

Biography

Laurent Verny is a PhD student in the Polymer Materials Engineering Laboratory (IMP) in the Université de Lyon (France). The unit has been divided in to four areas of excellence: Chemistry of Polymers, Structure and Rheology of Polymers: Process and Simulation, Physical Properties and Functional Materials, and Polymers at the Interface of Life Sciences. The IMP laboratory has wide fields of application such as polymers for energy, polymers for the car and aeronautical industries and polymers for medical applications. His research interests include reactive extrusion, polyimide and solvent free synthesis.

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