Investigation of Thermal Properties for Nylon-6 Obtained in a Polymerization Experimental Unit of a Batch Reactor

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Abstract – It is hard to overestimate the importance of nylon in the development of polymer science and in the commercial growth of polymer applications. Nylon’s combination of strength, toughness, and high melt temperature made it the first engineering thermoplastic, capable of myriad uses. The focus of this paper is to produce Nylon-6 in a batch reactor through hydrolytic polymerization of ε-caprolactam (monomer) and to compare thermal properties of the polymer obtained in the experimental unit with thermal properties of industrial Nylon-6 “grades”. The experimental unit was projected and implemented to study optimal operational conditions and produce polymers with desired properties. An experimental design can be performed aiming to obtain different “grades” of polymer.

In this work, an experimental unit of batch polymerization, that comprehends a polymerization reactor (autoclave), a nitrogen supplement system (cylinders), a vacuum system (vacuum pump), a water supplement system for refrigeration and cleaning, a polymer granulation system (granulator), and a drying system to polymer (vacuum greenhouse), in such way that nylon with specified properties may be achieved, has been developed and implemented.

The hydrolytic polymerization of ε-caprolactam is a very important commercial process and has drawn the attention of various researchers in recent years. In this process, the reactor feed typically contains fresh caprolactam monomer (CL), recycled monomer, water, and monofunctional acid as chain-terminating agent, such as acetic acid (AA). The reactor section hydrolyzes the monomer, converts it to a polymer, and builds up the polymer molecular weight. Because the molten polymer is in equilibrium with water and monomer, the byproduct water must be removed by vaporization to increase the molecular weight. However, the water vaporization may also remove a significant amount of caprolactam, which is recovered and recycled to the reactor. In conventional processes, the polymer is then pelletized and leached with hot water to remove low-molecular-weight extractables, such as residual monomer and cyclic oligomers. The polymer is then dried and shipped elsewhere.

Nylon-6 polymerized in experimental unit as well an industrial Nylon-6 “grade” have been analyzed through Differential Scanning Calorimetry (DSC), in equipment Mettler Toledo DSC 823e. Nylon-6 obtained in experimental reactor presented similar characteristics to industrial Nylon-6. Dynamic analyses were performed with heating rate at 10 °C/min. and temperature range from 25 to 300 °C. Samples were heated and cooled to obtain crystallization temperature. Analyses were performed. These results are shown in Figure 1. Different properties of Nylon-6 can be obtained through variation of process operational conditions.

![Figure 1: DSC analyses results for Nylon-6 produced in the experimental unit and industrial Nylon-6.](image)

References