

Some practical aspects of designing a laboratory scale batch polymerization reactor without gas entrapment and interfaced with virtual instrumentation

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Abstract

A 1-l stainless steel (SS) reactor with a glass bottom has been designed with symmetrically placed internals so that gas entrapment is completely avoided during bulk polymerization of methyl methacrylate (MMA). This reactor is instrumented and controlled using virtual instrumentation. The power consumed by a constant-speed stirrer motor is measured as a function of time which, along with the temperature history, can be used to infer the state (monomer conversion x_m and weight average molecular weight M_w) of the viscous reaction mass. This study highlights the importance of symmetry in the design of the reactor, proposes a novel reactor design, and discusses in detail the temperature control configuration that can help track any temperature history. Some preliminary results are reported for bulk polymerization of MMA and a correlation developed for the stirrer power, which can be used for soft sensing. © 2006 ISA—The Instrumentation, Systems, and Automation Society.

Keywords: Virtual instrumentation; Bulk polymerization; Batch reactor; On-line control; Flow visualization

1. Introduction

Most industrial reactors for the manufacture of commodity polymers operate under nonisothermal, batch, or semibatch reactor conditions. Initially, the temperature of the reaction mass is increased from the ambient temperature to the set point as fast as possible and then either an isothermal or a nonisothermal temperature history is imposed. There are three main challenges in this area, viz., the exothermic nature of the polymerization reaction, the viscosity of the reaction mass increasing with polymerization, and the nonavailability of *on-line* sensors for monitoring the state

variables (monomer conversion x_m and weight average molecular weight M_w) characterizing the system. The last of these has triggered the development of software sensors that can predict the properties of the reaction mass using secondary measurements, e.g., temperature, viscosity, and related variables (e.g., power input to a constant speed stirrer motor), heat removal from the reactor, etc. Chien and Penlidis [1] have presented an extensive review of on-line sensors for polymerization reactors. Unfortunately, most of these studies are reported for the control of *solution* polymerization reactors and not much information is available in the open literature on the control of free radical *bulk* polymerizations. The control of several bulk polymerization reactors, e.g., for styrene, methyl methacrylate (MMA), copolymeriza-

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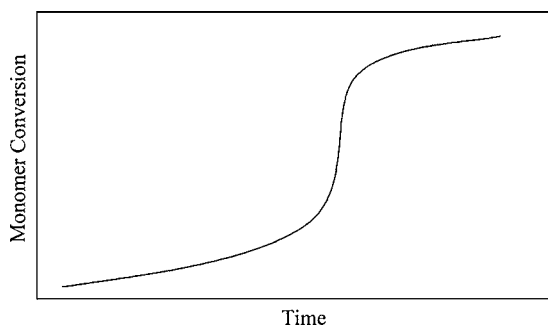


Fig. 1. Schematic representation of the Trommsdorff effect.

tions, etc., poses a formidable challenge, since these systems are associated with an extremely fast increase in the monomer conversion with time after the onset of the Trommsdorff or gel effect [2,3], as shown schematically in Fig. 1. There is very little control action that can be taken during or after this phenomenon is manifested, both because of its speed and because of the highly viscous nature of the reaction mass at this point. Model-based predictive control *before* the onset of the gel effect is the only alternative for such systems, where the objective is to obtain a product having the desired molecular weight distribution (MWD) and physical properties, even in the presence of disturbances (“save the batch”).

An important facet in on-line control of polymerization reactors is the on-line estimation of the state of the reaction mass. Jo and Bankoff [4] developed an on-line viscometer. They measured the variation of viscosity in a continuous flow stirred tank reactor (CSTR), in which solution polymerization of vinyl acetate in t-butanol was carried out. They used this to monitor the performance of the reactor. The feasibility of using measurements of the temperature $T(t)$ and viscosity $\eta(t)$ as a soft sensor, and its use for inferential control of bulk polymerizations, was demonstrated theoretically by Seth and Gupta [5], Bhargava Ram *et al.* [6], and Garg *et al.* [7]. Some exploratory experimental work has been carried out by Mankar and co-workers [8,9] using these measurements. All these workers used methyl methacrylate (MMA) as the sample system. It is more meaningful, however, to use $T(t)$, and the electrical power input $W(t)$, to a constant-speed stirrer motor [instead of the more fundamental variable $\eta(t)$] for on-line sensing. This is more relevant, industrially, since stirring is almost always present during polymerizations in batch or semibatch reactors, and $W(t)$ is, indeed,

related to $\eta(t)$. The use of these variables as soft sensors requires experimental data that can be used to develop appropriate correlations.

The objective of the present study is to develop an experimental, instrumented, and controlled (using virtual instrumentation), laboratory-scale batch reactor and take some data along these lines. The reactor should be able to track any desired (set point) temperature history $T_{sp}(t)$. Experimental data on $W(t)$ and $T(t)$ can then be taken under a variety of experimental conditions so that they can be used for on-line state estimation. It must be emphasized that $W(t)$ and viscosity can be correlated properly only if the reaction mass exists as a single liquid phase throughout the course of reaction. This is not easy to maintain. As the polymerization proceeds, the reaction mass becomes highly viscous and viscoelastic. Even a small amount of asymmetry in the reactor internals may lead to fluctuations at the surface of the reaction mass and induce entrapment of the inert gas (argon) present above the liquid. The high viscosity of the polymerization mass also results in poor removal of the exothermic heat of reaction, and results in vaporization of some monomer due to local overheating, again leading to entrapment of vapor and confounding the results on $W(t)$. An appropriate design of the reactor was finalized after several trials. Gas entrapment was avoided in this reactor throughout the course of reaction and under a variety of experimental conditions. This was confirmed by visual observation through a toughened glass window. The reactor was interfaced with a PC and controlled using LabVIEW™ (for set-point tracking). Some preliminary replicable experimental results on the polymerization of MMA are presented, under two temperature histories $T(t)$.

2. Reactor design

The final design of the reactor used in this study is shown schematically in Fig. 2. The head (top) of a 1-l stainless steel (SS) Parr™ reactor (Parr Instruments Company, Moline, IL, USA) was used. This houses an A1120HC magnetically coupled stirrer motor (1/8 HP, 130 V). Four SS tubes are also present (Fig. 2), attached, respectively, to a pressure indicator (PI), a rupture disc, a valve V_1 for pressurization, and a valve V_2 for purging. An SS cylindrical vessel with a supported-glass sys-

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