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Fine-Tuning of Process Conditions to Improve Product Uniformity of Polystyrene Particles Used for Wind Tunnel Velocimetry

by

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Background

Monodisperse polymer particles (having uniform diameter) have been used for the last two decades in physical, biological and chemical sciences. In NASA Langley Research Center monodisperse polystyrene particles are used in wind tunnel laser velocimeters. These polystyrene (P.S.) particles in latex form had been formulated at the Engineering Laboratory of the FENGD using emulsion-free emulsion polymerization. Monodisperse P.S. latices particles having different particle diameters had been formulated and useful experimental data involving effects of process conditions on particle size have been accumulated. However, similar process conditions and chemical recipes for polymerization of styrene monomer have often yielded monodisperse particles having varying diameters. The purpose of this summer research was to improve the P.S. latex product uniformity by fine-tuning of the process parameters based on knowledge of suspension and emulsion polymerization.

Present Study

A set of preliminary experimental runs based on a recommended process settings derived from previous studies made at NLRC revealed the need to: (1) monitor the temperature and PH of the reactor, agitator speed; (2) more complete cleaning of the reactor vessels to prevent seed polymerization in subsequent runs; (3) supplement the current approximate method of measuring particle size by Transmission Electron Microscopic (TEM) technique; and (4) measure the molecular weight and distribution of final particles to lend to better correlation of product properties with process parameters.

Runs made with these fine-tunings are shown on a table attached. The molecular weight data will be provided by the Polymer Laboratory at NLRC and the TEM measurements will be made by the author when he returns to his University. With the exception of four runs, the latex particle sizes of the runs can be represented as 2.0± 0.3 μm. Except for the run 294-3L the deviations of the particle size of the rest three runs can be attributed to premature stoppage of agitator due to undue build-up of polymer agglomerates and/or deviation of temperature by 2-3°C off the desired level of 65°C. Molecular weight and TEM data of the P.S. particles should shed more light on reasons for these deviations. The PH's of the latices were more or less the same.

Limited number of runs made to date at different agitator speeds (RPM) show that there is an optimum RPM range which yields particle size targeted. An agitator speed below this range causes reduced particle size and speed above this range would cause mechanical coalescence of the particles yielding large agglomerates. Further runs to investigate this is in progress.

A set of recommendations for fine tuning of the process parameters to improve P.S. latex product uniformity is transmitted to the Engineering Laboratory of FENGD.

EMULSION-FREE STYRENE POLYMERIZATION PROCESS DATA

Run No.	Temp Initial/Final °C	Agitator RPM Initial/Final	D/I Water M Ω cm	PH (Final)	% Solid	Dp (μm) (Nikon Micrograph)	Dp*** (TEM)	***MW (GPC)
290-3L	62.8/65.1	150/Stopped	10.0	-	6.0	1.7		
291-3L	64.5/65.2	150/Stopped	7.0	2.6	6.2	2.0		
292-3L	62.8/65.4	150/Stopped	7.0	2.6	6.36	2.1		
293-3L	62.5/64.7	150/120	7.0	2.6	6.3	2.3		
294-3L	62.9/64.1	150/140	18.0	2.65	6.14	1.0		
295-3L	62.5/65.8	150/150	18.0	2.60	6.45	2.1		
296-3L	63.0/64.6	150/100	18.0	2.65	6.0	2.1		
*297-3L	62.6/62.8	150/50	18.0	2.75	5.61	2.7		
**298-3L	63.0/65.3	150/120	18.0	2.60	-	2.2		
**300-3L	62.3/62.3	150/125	18.0	2.6	5.73	2.6		
**301-3L	62.3/65.2	150/Stopped	18.0		6.0	1.3		

** Reactor Heater and Agitator Motor Connected to Constant Voltage Source

* 62 ml of 1% $MgSO_4$ Used Instead of Usual 55 ml

*** To be Analyzed at LaRC Polymer Laboratory and Christian Brothers University