

Removal of Residual Monomers and Solvents from Granular Polymers in Fluidized Beds; Experimental Methods of Measurement of Diffusivity and Solubility Parameters for Design and Troubleshooting

Manuk Colakyan, The Dow Chemical Company, P.O. Box 8361, South Charleston, WV 25303 and **Glenn W. Baldwin**, R&D, The Dow Chemical Company, P.O. Box 8361, South Charleston, WV 25303.

Various technologies available for the removal of monomers from granular polymers will be discussed. Emphasis will be given to fluidized bed operations and how to model and scale-up such contactors. Techniques for the measurement of diffusion coefficients of monomers from polymers, as well as the measurement of solubility parameters will also be presented.

Residual solvents and monomers contained in polymers are removed to improve product safety, meet customer requirements and eliminate or help reduce emissions. The process of removing the residual organics from polymers is sometimes coined as “purging” and can be carried out in various contactors where an inert gas or air flows through a vessel containing either a batch of the polymer or polymer flowing through it. In this paper we limit our presentation to gas-solid systems. The contacting operation can for example, be done a) in a moving packed bed contactor where a “purge” gas flows counter-currently through it; b) a batch of stagnant solids where the “purge” gas flows through it; or c) a batch or continuous fluidized bed, where the fluidizing gas is the “purge gas”. If we limit these operations to solvents or monomers that are not present as “free moisture”, the underlying physical phenomena for the transport of the solvents from the particle to the gas is common to all, i.e., the transport of the solvent or monomers by diffusion from granular polymer to its surface and the convective removal of the solvents or monomers by the gas. In this presentation we focus on fluidized bed operations.

Colakyan and Eisinger (1) present the theoretical background to model the removal of residual monomers in fluidized beds. We will briefly summarize their findings. The models used in predicting the removal of monomers or solvents from granular particles use two key parameters for estimations, namely the diffusion coefficient of the solvent from the polymer and its solubility. The latter can be conveniently expressed by using Henry’s Law. In this presentation we will focus on the techniques for measuring the Henry’s Law constants and diffusion coefficients. Determination of Henry’s constant (H) of a solute in a polymer requires measuring the amount of the solute dissolved at equilibrium in the polymer at a given temperature and partial pressure of the solute. This can be done by using two techniques: a) by weighing a sample without direct mechanical linkage between the balance and the sample, a magnetic suspension balance could serve this purpose; b) by using a headspace GC. The measurement of the diffusion coefficient involves a dynamic experiment which helps determine the rate of removal of the solvent in time. There are several methods for this measurement. These methods either use the direct measurement of monomer concentration remaining in the polymer by analyzing the amount of solvent contained in the monomer, or back-calculating the solvent remaining in the polymer by measuring the concentration of the solvent in the gas which contacts

the polymer in real time. There are several types of experimental devices which can be used for carrying out the diffusion measurements. Each offers varying degrees of complexity for the estimation of the diffusion coefficient. We will present methodology for obtaining the diffusion coefficients from three devices: a) a shallow fluidized bed; b) the above mentioned magnetic suspension balance; c) a moving packed bed of solids with counter-current “purge” gas flowing through it. Advantages and disadvantages of each technique will be discussed.

1) Colakyan, M.; Eisinger, R. S., Removal of Residual Monomers from Polymers in Fluidized Beds
Ind. Eng. Chem. Res.; **2003**; 42(12); 2654-2660