

Chapter 9

Conclusions

9.1 The extruder as a polymerisation reactor

The counterrotating twin screw extruder can be used as a polymerisation reactor for the synthesis of polystyrene, styrene-*n*-butylmethacrylate-copolymer and several related polymer blends. In one step, a conversion of 95 to 99% is possible, when styrene (St) or styrene-*n*-butylmethacrylate (St-BMA) is polymerised by means of a radical mechanism. To obtain this conversion, a high die resistance is necessary, as a result of which the throughput is limited. Besides, the throughput is limited by the large viscosity differences of the material inside the extruder when it travels from feedzone to die end. Therefore, the throughput is more or less a variable rather than a primary parameter.

The working domain of the extruder is defined as the range of the extruder parameters in which a sufficient conversion is obtained in a stable process. Experiments in this thesis showed that this operating window is strongly influenced by material parameters, especially polymerisation rate and polymer melt viscosity.

Prepolymerisation enlarges the working domain due to the reduced reaction time and increased feed viscosity. Therefore prepolymerisation is, especially in the case of St or St-BMA, highly advisable.

Crucial in explaining the results is the question whether or not a plug of completely reacted material is present before the die. If this plug is not present, the required conversion will not be achieved. A large plug leads to sufficient conversion, but the extruder volume is not used economically in that situation. Besides, the energy input is unnecessary high. However, for a stable process, a certain plug seems necessary. The die resistance is the most powerful tool for creating a plug of completely reacted material.

The theory, developed in this study, is very useful in optimising the extruder when it is used as a polymerisation reactor.

9.2 Conclusions concerning product properties

The extruder can be used for synthesising polymers of St and St-BMA with a weight average molecular weight (M_w) that ranges from 30 to 200 kg/mol. This range makes the extruder promising for synthesising either materials, which can be used for resin additives as well as for engineering plastics. The molecular weight is strongly influenced by initiator concentration and to a lesser degree by the barrel temperature. Other extruder parameters

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such as screw rotation rate and die resistance hardly influence the molecular weight or the influence is unpredictable. A faster polymerisation results in a higher molecular weight. The effect of the chain transfer agent *t*-dodecylmercaptane is remarkably small in the case of St.

Slight crosslinking is possible by means of reactive extrusion. Via reactive extrusion, polymers with a gel fraction of around 60% can be easily obtained. A very small amount of crosslink agent is a method to increase the molecular weight of the polymer, without obtaining a non-soluble gelfraction.

Reactive extrusion is also a useful tool for synthesising polymer blends. The polymerisation of St in which PPE (poly-2,6-dimethyl-1,4-phenylene oxide) is dissolved yields a transparent polymer blend, which indicates complete mixing of the polymers. The addition of a rubber component (SBS) results also in a fine dispersion of the polymer in the newly formed matrix. However, due to this fine dispersion, the improvement in impact strength is rather poor.

9.3 Conclusions concerning the theory of the extruder

The experiments in chapter 2 show that, although the counterrotating twin screw extruder has been studied quite often, a predictive model for the residence time distribution (RTD) for this type of extruder is not available yet. This study showed that a good prediction of the mean residence time can be obtained by multiplying the number of fully filled chambers that is predicted by theory by a factor 1.3 and by taking into account the total non-pressure driven leakage flows in the partially filled zone.

For describing the RTD of an extruder, the theory described by Chen (1993, see chapter 2) seems very promising. This theory calculates the RTD of the extruder by using the RTD of one single chamber. However, for a good estimation of the RTD of one C-shaped chamber, three-dimensional flow simulations in combination with particle tracking should be used. The method as described by Chen can also be useful for modelling the extruder as a polymerisation reactor. However, since the throughput is not a parameter with a defined value in reactive extrusion, the modelling of the extruder as a polymerisation reactor will consume a lot of computing time.