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The investigation of polymerization process by means of ultrasound

1. Introduction

Ultrasound is wide used in physical measuring process as well as in technological applications. The advantages of the ultrasound are as follows: optical transparency of the medium is not required, transmission is easily realizable by power variation at the transducer and a fast electronical signal processing is possible. The measuring values are velocity c and absorption coefficient α of the ultrasound. Both are close related to the microscopie structure of the substance studied. Because the velocity can be relatively easily examined with a high accuracy (10^{-5} or better) this parameter is well suitable for the process control.

Hitherto, the attenuation has only been measured during the polymerization process [1—3]. Since the absorption is very sensitively influenced by scattering of sound, the absorption method is preferentially applied to such processes where gaseous phases appear, for instance to the vinyl chloride polymerization

[2, 3]. On the other hand, the velocity method will be useful to monitor reactions which are going on without formation of a foam phase.

The aim of this paper is to show that measuring the ultrasonic velocity during the polymerization process can provide information about the actual state of the reaction.

2. *Model consideration*

In a polymerizing system which contains several components the ultrasound velocity is a function of these components and of temperature T. Choosing a reference substance whose ultrasonic velocity does not change during the process or varies in a known manner, the variation of c due to the component i can be described as follows

$$(\Delta c)_i = k_i \left(\frac{\Delta c}{\Delta k}\right)_i, \tag{1}$$

where k_i is the concentration of the ith component within the reference substance and $\left(\frac{\Delta c}{\Delta k}\right)_i$ is the corresponding sound-concentration-coefficient. Only in a few exceptional cases it is possible to add the sound velocities of the several components to a resulting velocity of the whole system. But, if we know the coefficient $\left(\frac{\Delta c}{\Delta k}\right)_i$ of each component as a function of the concentration k_i and the temperature the resulting velocity can be written by

$$c = c_0 + \sum_i k_i \left(\frac{\Delta c}{\Delta k}\right)_i, \tag{2}$$

where c_0 is the velocity in the reference substance. After experimental determining the coefficient $\left(\frac{\Delta c}{\Delta k}\right)_i$ the concentration of a component k_i can be calculated from the velocity c measured experimentally. Thus, it is possible to decide the polymer conversion during the polymerization. In the case of emulsion and suspension polymerization the equation (2) takes the form

$$c = c_0 + k_{P+S}\left(\frac{\Delta c}{\Delta k}\right)_{P+S} + k_{LM}\left(\frac{\Delta c}{\Delta k}\right)_{M<S} + (k_M{}^{\text{total}} - k_{LM})\left(\frac{\Delta c}{\Delta k}\right)_{M>S} + k_I\left(\frac{\Delta c}{\Delta k}\right)_I + k_E\left(\frac{\Delta c}{\Delta k}\right)_E \tag{3}$$

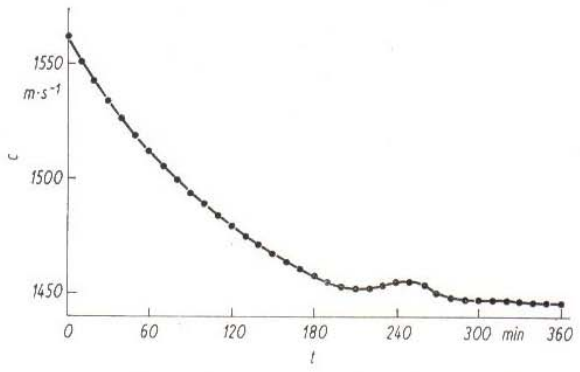

Fig. 1. Velocity c as a function of time for emulsion polymerization of VAC

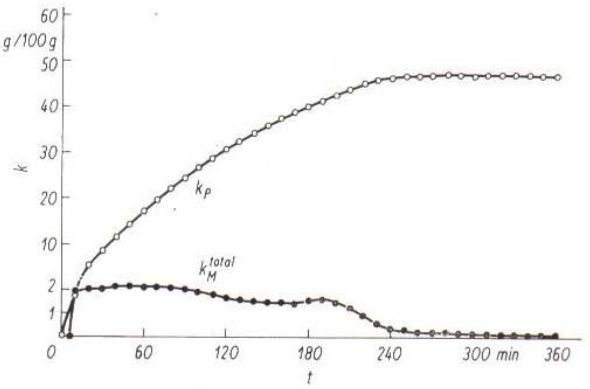

Fig. 2. Monomer and polymer concentration as a function of polymerization time

where k_{P+S} is the concentration of the polymer and the stabilizator, k_{LM} is the limit concentration of the dissolved monomer, $k_M{}^{\text{total}}$ is the total concentration of the monomer, k_I the concentration of the initiator, and k_E that of the electrolyte. $M < S$ means the region of the solubility of the monomer and $M > S$ that outside of the solubility.

3. *Experimental results*

Using the ultrasonic pulse method [4] at a frequency of 800 kHz and with fixed distance (10 cm) of two transducers, the emulsion polymerization of vinyl acetate in presence of emulgator has been investigated as a semicontinuous procedure within a two liters autoclave. The ultrasound velocity c was measured with an accuracy of 1‰. The velocity as a function of polymerization time is shown in Figure 1. The polymer and monomer concentration calculated after Equ. (3) are given in Figure 2. These experimental results confirm that the change of ultrasonic velocity during the polymerization characterizes the actual state of the process.

4. *Conclusions*

The basic principles of a ultrasound technique to study reaction process have been described. Our results concerning the vinyl acetate polymerization show that one can immediately get quantitative informations about the polymer conversion and the kinetics of the process. Furthermore, this method is well suitable for the process regulation.

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F. DINGER, P. HAUPTMANN, R. SÄUBERLICH und S. WARTEWIG

Technische Hochschule „Carl Schorlemmer" Leuna—Merseburg, Sektion Physik, Wissenschaftsbereich Experimentalphysik II, DDR-4200 Merseburg

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