7. Investigation of Mixing with the Test Reactions in a Semi-Batch Reactor.

7.1. Experimental System and Experimental Procedure.

Experiments were conducted in a cylindrical vessel made of glass (figure 7.1). The reactor was equipped with four baffles and a four blade pitched-blade turbine pumping downwards. The baffles were spaced 90 degrees one from another. The blades of the turbine were 1 mm thick and inclined 45 degrees from the turbine shaft. The turbine was installed centrally in the mixer. The diameter of the turbine and the distance between the reactor bottom and the turbine were equal to one third of the tank diameter. In order to avoid deviations of the turbine shaft from the mixer axis, its lower tip was mounted in a bearing attached to the reactor bottom. The turbine was driven by a variable speed DC motor equipped with an optical revolution counter. The turbine, its shaft, the baffles and the bearing were made from stainless steel. The reactor was fed from a small feeding pipe made of glass and mounted vertically between the baffles. The internal diameter of the outlet of the dosing pipe was equal to 1.6 mm and the feeding point was placed 6 mm above the turbine. The pipe was connected with a silicon tube to a syringe pump. The reactor was placed in a thermostat-

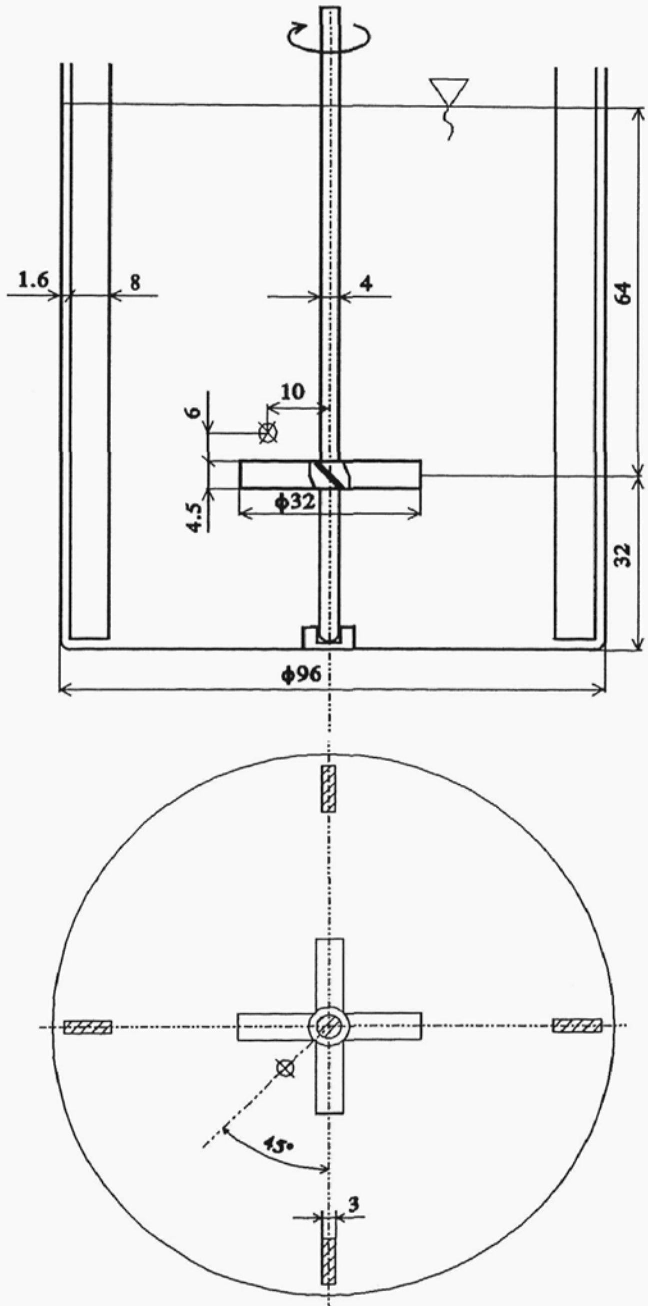

Figure 7.1. Scheme of a semi-batch reactor.

ing bath. The major part of 1m long silicon tube having 3 mm internal diameter and connecting the metering pipe with the syringe pump was immersed in the bath. Thus, the temperature of the solution pumped through the connecting tube into the reactor was the same as the temperature of the reactor content for feeding rates not higher than 2 cm^3/min. The temperature of the thermostating bath was equal to 25°C.

The solutions mixed in the reactor were composed of: water, polyethylenepolypropylene glycol as the viscosity increasing agent, potassium chloride as the density increasing agent and substrates of the competitive-parallel reactions (6.1). The viscosities and densities of the substrates solutions and of the resulting mixture were always measured at the same temperature at which tests were performed. Before each experiment concentrations of sodium hydroxide and hydrochloric acid in their solutions were determined by pH-titration. The concentration of acid was determined prior to addition of ester and afterward recalculated for the final solution volume. The initial and final concentrations of ethyl chloroacetate in the mixer content were determined by HPLC (see chapter 6.5).

The chemically equivalent amounts of acid, base and ester were used in experiments; the reactant initial concentrations, they would have had if the whole quantity of base had been suddenly added to the reactor and completely mixed with its content, were equal to 0.01 mol/dm^3. At the beginning of each experiment the weighted amount of the acid and ester solution was poured in the reactor. Then rotation of the pitched-blade turbine was initiated and after setting its speed at a desired level and stabilizing the temperature of the mixer content, dosing of the base solution into the reactor was started. The flow in the reactor was laminar; the stirrer Reynolds number:

$$Re = n \cdot d^2 \cdot \rho / \mu \qquad \textbf{(7.1)}$$

where **n** is agitation speed and **d** is the turbine diameter, was smaller than 24.

The volumes of mixed solutions were chosen in such a way that after metering half of the base solution, the liquid depth was equal to the diameter of the reactor. When the feeding was completed, the rotation speed of the turbine was kept unchanged for at least half an hour if it was greater than 200[rev/min], and an hour if it was smaller or equal than 200[rev/min]. Finally, before taking samples for HPLC analysis, the whole reactor content was homogenized by means of a propeller having a diameter equal to 70mm.

The final selectivity in the tests was computed from the following expression:

$$X = \Delta N_{ester} / N_{NaOH} , \qquad \textbf{(7.2)}$$

where ΔN_{ester} is the number of moles of ethyl chloroacetate hydrolysed in reaction with the sodium hydroxide and N_{NaOH} is the number of moles of base added to the reactor and consumed in reaction with acid and ester.

7.2. Experimental Results.

7.2.1. Effect of the Feeding Time on the Product Distribution.

The feeding time can influence the product distribution in two ways. Very fast feeding can produce high concentration gradients on the macroscopic scale (comparable with the reactor size). Inhomogeneity on the macroscale retards mixing on the molecular scale (high diffusion times) and consequently elevates the final selectivity. On the other hand, in the case of very slow feeding the initial size of the inlet stream can be very small. This results in faster diffusional mixing, which decreases the final selectivity. As it can be seen, it may be impossible, even in the case of very slow feeding, to avoid influence of the feeding time on the product distribution.

Two series of experiments were conducted in the semi-batch reactor to determine the effect of the feeding rate on the final selectivity. In the first series the agitation speed was equal to 100 rev/min, whereas in the second series the agitation speed was equal to 400 rev/min. The viscosities and densities of mixed solutions were kept constant and almost equal to each other. The initial volume ratio $\mathbf{a=V_{HCl+ester}/V_{NaOH}}$ was equal to 19.

Tables 7.Iabc and 7.IIabc show compositions, volumes, viscosities and densities of the solutions together with feeding rates $\mathbf{Q_f}$ and final selectivities of the reaction. Figure 7.2 presents the final selectivities plotted versus the feeding time.

Table 7.Ia. First series of tests - initial reactor content; $\mathbf{w_p=40\%}$.

Exp.no.	HCl [mol/dm^3]	Ester [mol/dm^3]	V [cm^3]	ρ [g/cm^3]	μ [Pa·s]	KCl [g/kg]
1	0.01012	0.01024	680.10	1.0680	0.306	11.50
2	0.01029	0.01030	680.04	1.0684	0.307	11.56
3	0.01012	0.01024	680.10	1.0680	0.306	11.50
4	0.01025	0.01056	680.04	1.0681	0.303	11.50
5	0.01025	0.01056	680.04	1.0681	0.303	11.50
6	0.01013	0.01066	680.17	1.0679	0.305	11.50