

## VisiMix TURBULENT. BATCH REACTOR SCALING – UP.

### Introduction.

This example is based on a practical case application of VisiMix Turbulent by one of our clients. It is related to development of a new process of organic synthesis in a chemical plant that belongs to one of the big international pharmaceutical corporations. One of the stages of this synthesis - a relatively fast chemical reaction between two reactants accompanied with a side reaction.

**Schematically: the main reaction as  $A + B > C$  with the side reaction as  $B+B > D$ .**

On the development stage, a low quantity of by-product and acceptable degree of purity of the main product was obtained in a batch pilot reactor (total volume - about 2 liter).

The process was transferred to a production glass lined 1230 liter reactor.

**Scaling-up was based on reproduction of mixing time value. However, a significant deterioration of the product purity and increase of by-product concentration occurred.**

VisiMix program was used by the pilot department engineers for identifying the problem and finding the way to reproduce the product quality.

### Problem description:

It can be expected from the data on reaction presented above, that yield of the by-product can be reduced if the reaction is provided at a lower concentration of the reactant B. If distribution of the reactant B in the reactor is not uniform, **the by-product formation is expected to be the most active in locations and within the period of time that correspond to the maximum values of the B concentration.**

The VisiMix program is used below for comparison of these parameters in the pilot and production reactors. For this purpose we will perform mathematical simulation of chemical reaction in these reactors using accessible initial data, and compare **maximum values of reactant B** and the periods of time necessary for the reactant distribution inside the reactor.

*NOTE: Approximately physical properties of solutions and reaction kinetics data can be accepted for such calculations. Exactitude of the assumed values does not influence the essential results and conclusions.*

### Initial data:

Laboratory reactor.

Solution of the reactant A (about 300 ml) was loaded in the reactor. The reaction was provided by instant adding of reactant B solution (about 100 ml) through a tube of about 5 mm diameter, fixed on the distance 10 mm from the wall and 65 mm from the bottom.

Total quantities of the reactants A and B per test – 0.8 g-mol.

Mixing in the pilot reactor was performed with agitator of the ‘Glass lined impellers, Impeller 1’ type (see VisiMix Impeller types, Figure 5).

Physical properties of solution are close to physical properties of water.

**Technical characteristics of the reactor:**

Tank with elliptic bottom:

Inside diameter = 100 mm;

Total tank height = 225 mm;

Let us create a project **R-PILOT. vsm**. Select a **tank with elliptic bottom** without jacket in the arriving Tank types selection window (Figure 1) and enter the data above (Figure 2).

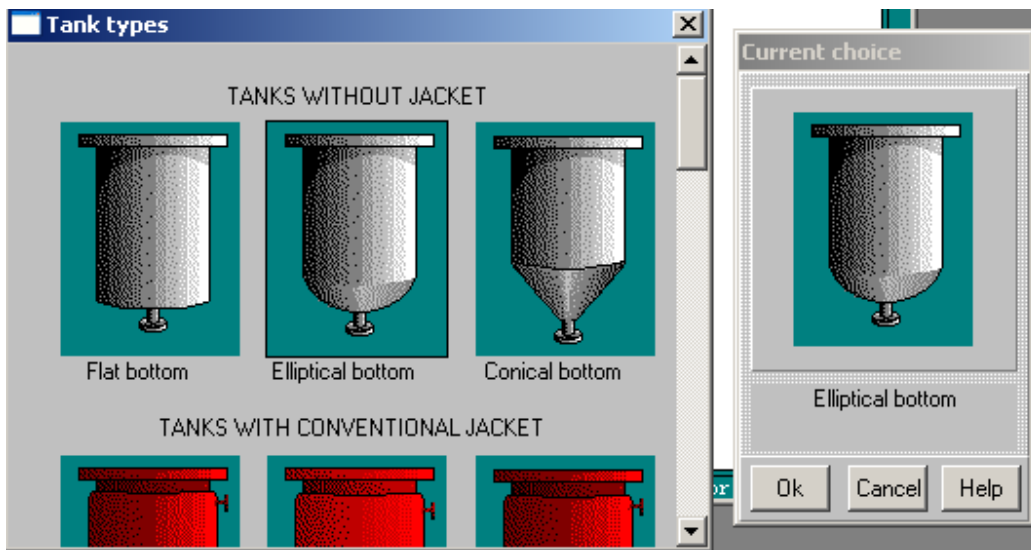


Figure 1. VisiMix graphic tanks selection.

**TANK WITH ELLIPTICAL BOTTOM**

Inside diameter	100	mm	▼
Total tank height	225	mm	▼
Total volume	1.70	l	▼
Level of media	59.26	mm	▼
Volume of media	0.4	l	▼

**Figure 2. Entering Tank data for the pilot reactor.**

After the table has been completed, click anywhere on the field of the window, and the tank diagram on the screen will change to reflect your input. Click **OK** to confirm your input. The **Baffle types** menu will now appear.

### Baffles:

The 5 mm pipe mentioned above can be entered as a **Beavertile baffle**. Click on the diagram of the selected baffle type and it will appear in the **Current choice** window on the right (Figure 3). Click **OK** to confirm the choice. Enter parameters of your baffle in the table that appears (Figure 4).

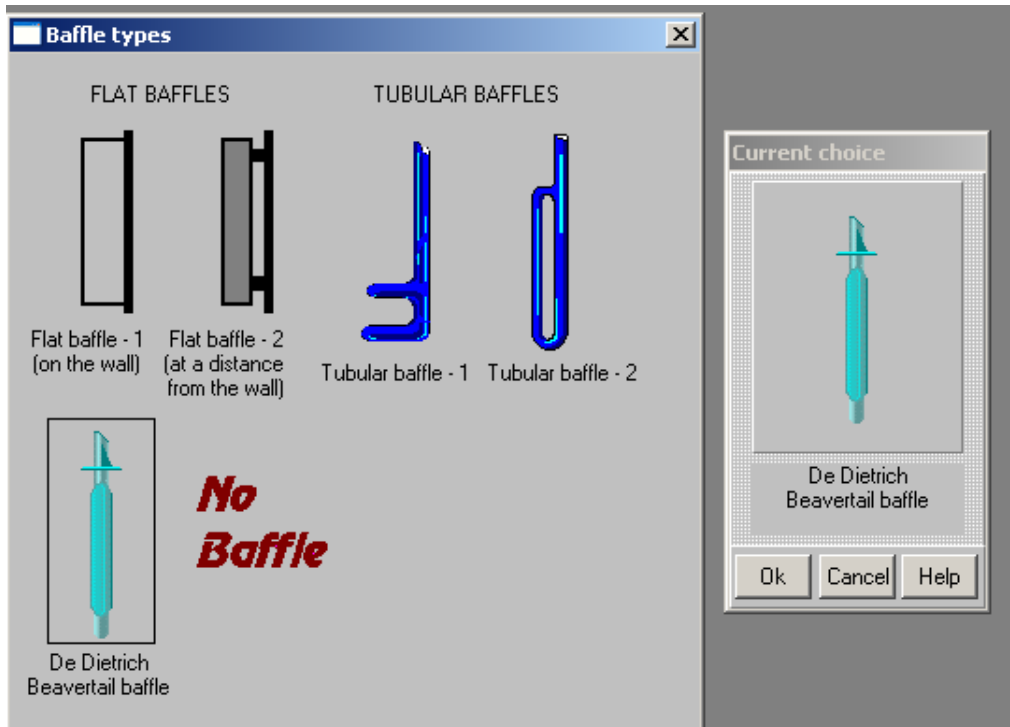


Figure 3. VisiMix graphic baffles selection.

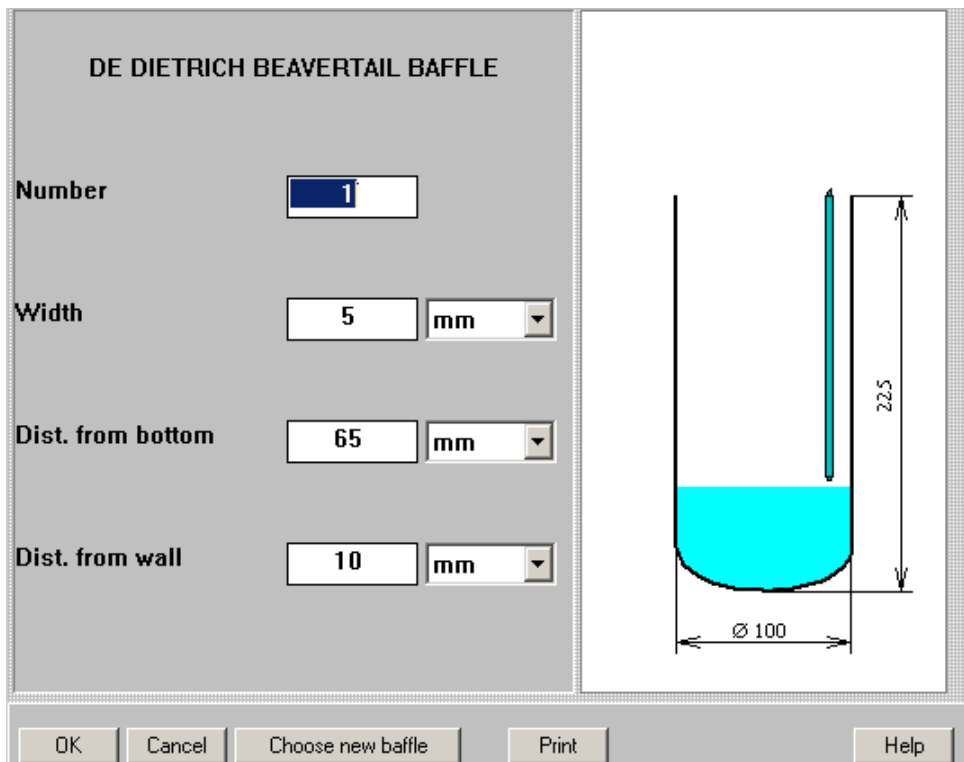


Figure 4. Entering baffle data for the pilot reactor.

Confirm data with OK, and the Impeller types selection window will appear automatically.

Mixing device :

Tubular impeller with flatten back-wended blades .

Tip diameter = 60 mm;

Number of blades = 3;

Width of blade = 3 mm;

Distance from bottom = 20 mm;

RPM = 250;

Power of drive = 100 W;

Select the **Glass lined impeller-1** in **Impeller types** selection window (Figure 5), and it will arrive in the **Current choice** window. The **single impeller** option (for single-stage mixing device) is already shown (by default). The impeller parameters – sizes, position and rotation speed – are entered in the next input table that appears automatically (Figure 6). After the data are entered, click anywhere in the field of the window, and the diagram on the screen will change to reflect the last input. Click **OK** to confirm the input.

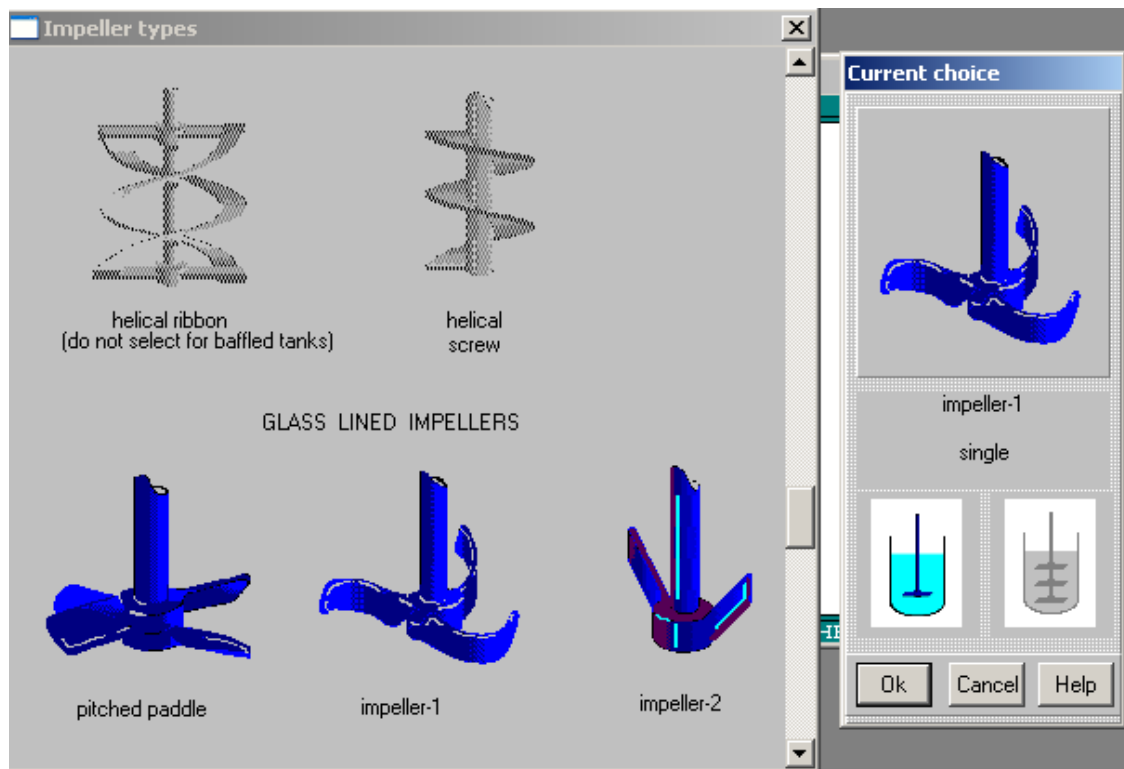


Figure 5. VisiMix graphic Impeller types selection.

**GLASS LINED IMPELLER 1**

Tip diameter:  mm

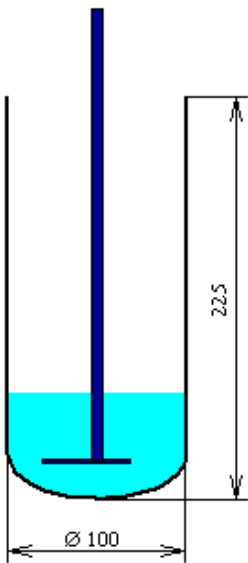
Number of blades:

Width of blade:  mm

Dist. from bottom:  mm

Rotational speed:  Rpm

Motor power:  KW



OK Cancel Choose new impeller Print Help

Figure 6. Entering baffle data for the pilot reactor.

You will now be requested to enter properties of the media in AVERAGE PROPERTIES OF MEDIA input table. It is supposed that they are close to properties of water (Figure 7).

**AVERAGE PROPERTIES OF MEDIA**

Type of media:  Newtonian  Non-Newtonian

Average density:  kg/cub.m

Dynamic viscosity:  cP

Kinematic viscosity:  sq.m/s

Constant K:  Pa\*(sec)<sup>n</sup>

Exponent n:

Yield stress:  N/sq.m

Behavior of Non-Newtonian media is approximated with the functions:

$$\tau = \tau_0 + K * \gamma^n$$

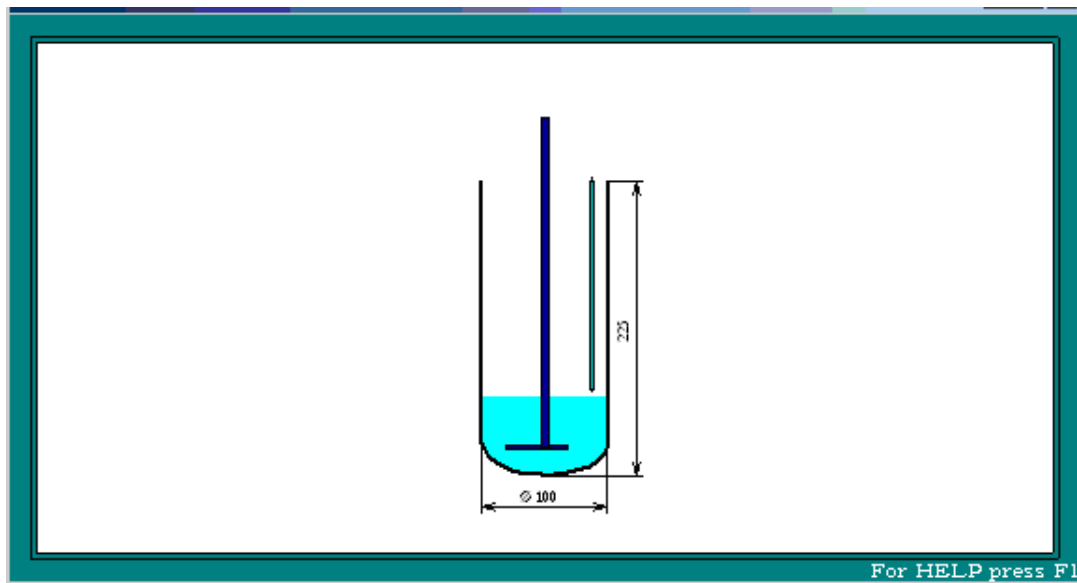
$$\mu = \tau_0 * \gamma^{-1} + K * \gamma^{n-1},$$

where  $\mu$  - dynamic viscosity, Pa\*sec;  
 $\gamma$  - shear rate, 1/sec;  
 $\tau$  - shear stress, Pa;  
 $\tau_0$  - yield stress, Pa.

OK Cancel Print Help

Figure 7. Entering average properties of media.

After we have entered basic data for the pilot reactor, its diagram appears (Figure 8).



**Figure 8. Scheme of the pilot reactor.**

Now let us create a project corresponding to the Production reactor.

### Production reactor.

Solution of the reactant A (about 525 l) is loaded in a glass lined 1200 liter reactor (Pfaudler) with a tubular baffle (glass lined). The reaction is provided by instant adding of reactant B solution (about 175 l) on the surface of the liquid using a single tube. Distance from the wall – about 100 mm. Total quantities of the reactants A and B per batch – 105 g-mol.

Mixing in the reactor is performed with a Glass lined Impeller 1 (Pfaudler type).

### Technical characteristics of the reactor:

#### Tank with elliptical bottom:

Inside diameter = 1200 mm;

Total tank height = 1190 mm;

#### Impeller characteristics:

Tip diameter = 900 mm;

Number of blades = 3;

Width of blade = 133 mm;

Distance from bottom = 225 mm;

RPM = 50;

Power of drive = 3300 W;

Characteristics of the baffle:

Type – tubular baffle-2;

Number = 1;

Tube diameter = 3.6 in;

Distance from bottom = 330 mm;

Distance from wall = 59 mm.

It is assumed that physical properties of media in the two reactors are identical, and only size and design is different. So, we can save the existing project as **R-PRODUCTION.vsm** and change the equipment characteristics – Tank, Baffles and Impeller – accordingly to the technical characteristics of the production scale tank presented above (Figures 9-12).

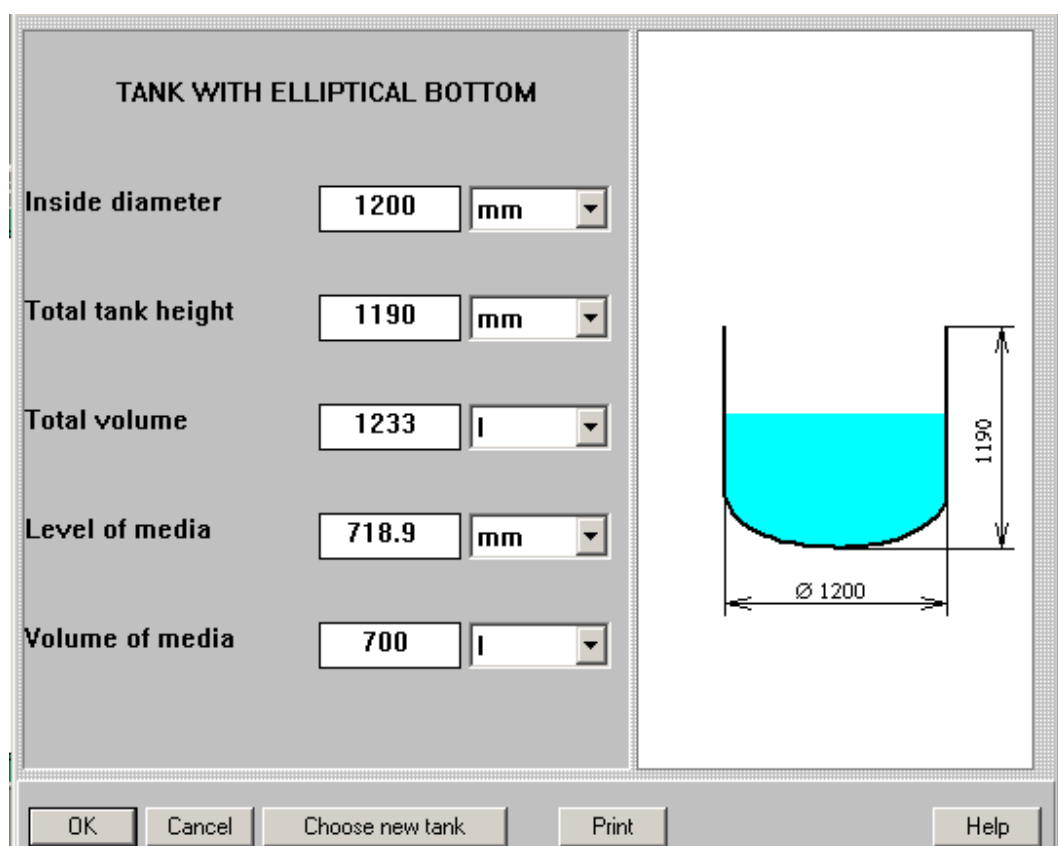


Figure 9. Tank data for the production reactor.



**TUBULAR BAFFLE-2**

Number

Tube diameter (D)  in

Length (L)  mm

Dist. from bottom  mm

Dist. from wall  mm

Angle to radius (fi)  deg

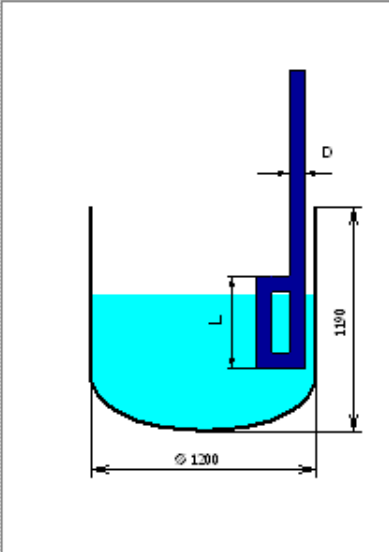
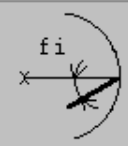



Figure 10. Baffle data for the production reactor.

**GLASS LINED IMPELLER 1**

Tip diameter  mm

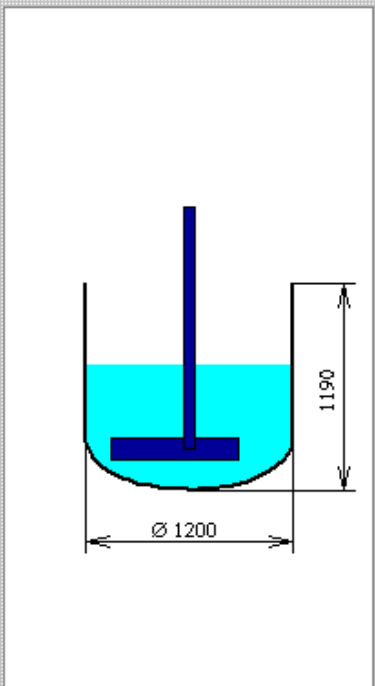
Number of blades

Width of blade  mm

Dist. from bottom  mm

Rotational speed  Rpm

Motor power  KW



OK Cancel Choose new impeller Print Help

Figure 11. Impeller data for the production reactor.

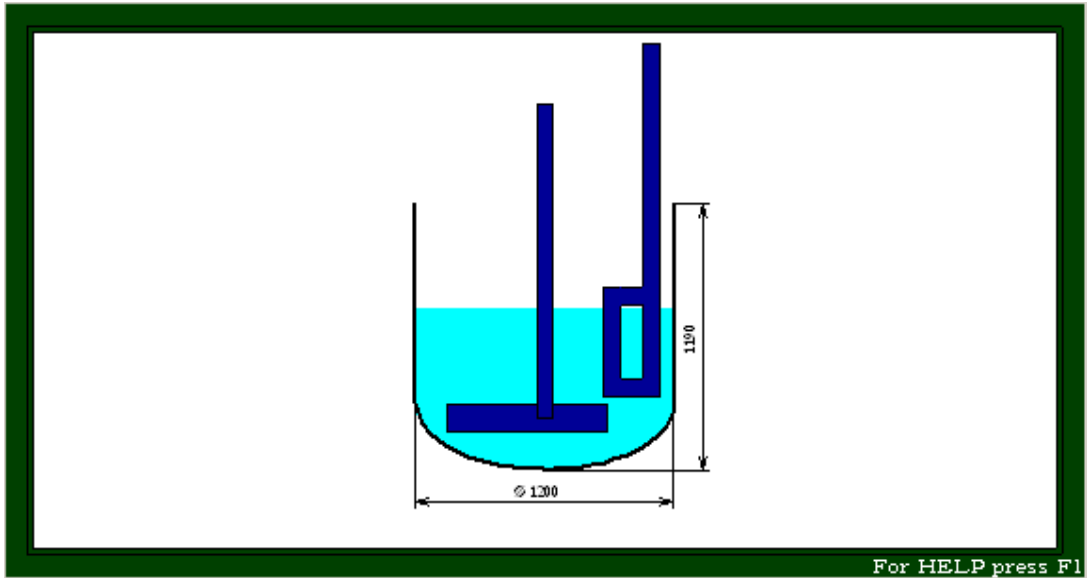


Figure 12. Scheme of the production reactor.

### Calculations and comparison of the reactors.

From now on, we can start calculations and compare mixing and mixing-dependent parameters of the two reactors. As the first step, let us compare the parameter that has been used by process developers as a base for scaling up – **mixing time**.

Mixing time.

Let us close all windows (via **Window>Close all**), select **Project list** in the **View** sub-menu and activate the project **R-Pilot.vsm** corresponding to the pilot reactor. The scheme of the reactor (Figure 8) arrives in the screen.

Click **Calculate** and select **Single-phase liquid mixing**.

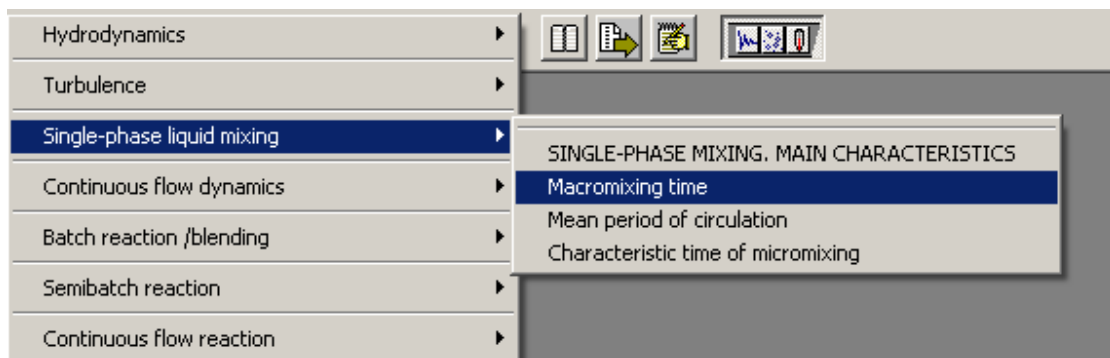


Figure 13. Menu Single-phase liquid mixing.

Now select **Macromixing time** in the arriving sub-menu (Figure 13). In response, the program performs simulation of the macroscale tracer distribution returns calculated value of the requested parameter (Figure 14).

MACROMIXING TIME		
Parameter name	Units	Value
Macromixing time	s	13.5

For HELP press F1

Figure 14. Mixing time in the pilot reactor.

Now let us activate the project **R-Production.vsm** the production reactor (via **View>Project list**). The production reactor scheme (Figure 12) will arrive. In order to repeat the modeling and define the same parameter for this reactor, click Last menu in the main menu bar (Figure 15).

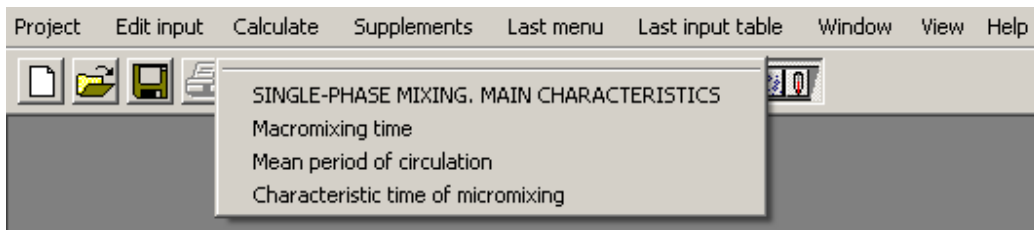


Figure 15. Menu Single-phase liquid mixing as the Last menu option.

Now select Macromixing time in this menu. The corresponding table for the production reactor will arrive. Comparison of the Macromixing time output table for the two reactors is shown in the Figure 16 (via **Window>Tile** in the main menu bar).

**The comparison shows that mixing time values for the two reactors really are practically the same, the difference of the two values is not statistically significant.**

[R-PILOT] - Macromixing time			[R-PRODUCTION] - Macromixing time		
MACROMIXING TIME			MACROMIXING TIME		
Parameter name	Units	Value	Parameter name	Units	Value
Macromixing time	s	13.5	Macromixing time	s	11.7

Figure 16. Mixing time values for the production and pilot reactor.

So, our next step – mathematical modeling of the bath reactor and comparison of maximum degree of non-uniformity of the reactant B distribution.

Let us click **Batch reaction / blending** in **Calculate** menu (Figure 17) and select **Max. local concentration of reactant B, batch reactor**.

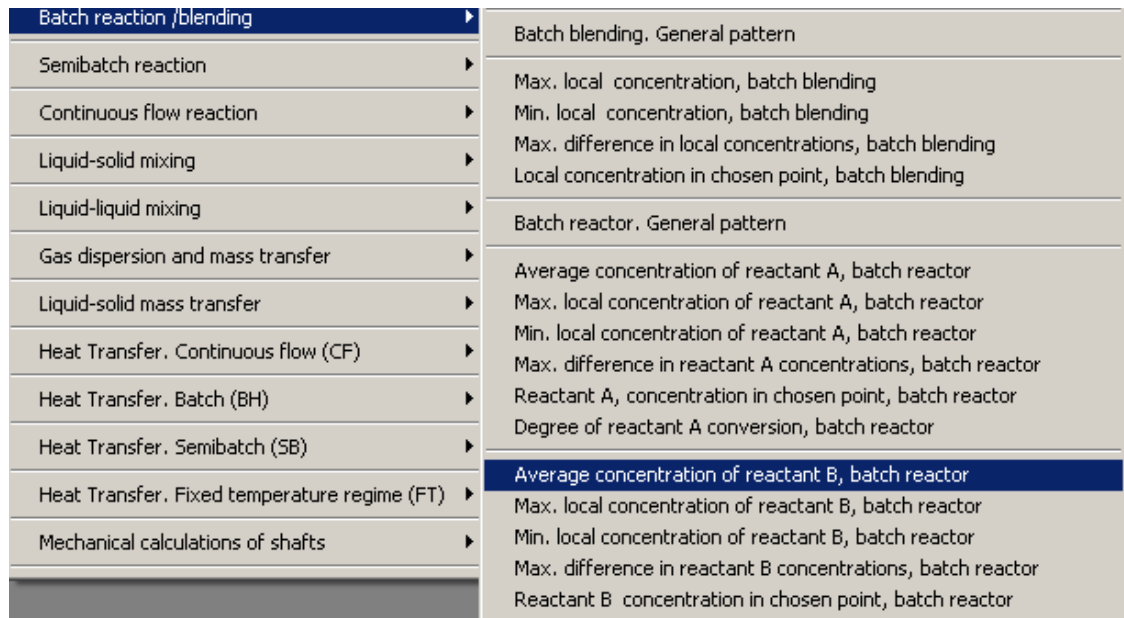
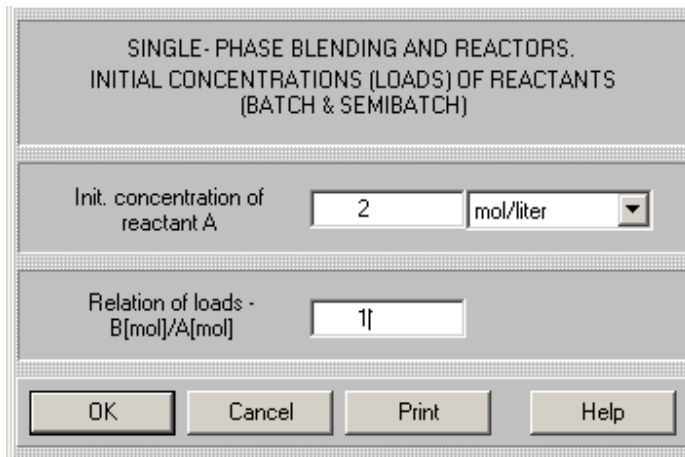


Figure 17. Batch reaction menu.

In this point the program provides us with additional input tables, and we have to enter the specific data necessary for mathematical modeling of batch reactors - kinetics of the main and side reaction and concentrations of the reactants (Figures 18 and 19). The reaction kinetics has not been studied quantitatively, and specific reaction rates of the main and side reactions are not known. Therefore comparison of the two reactors on this stage is performed using the 'FAST REACTION' approximation, by entering **F** as the **Specific reaction rate** for the main reaction (Figure 18). In this case the side reaction is neglected.

Figure 18. Entering data for the FAST REACTION approximation.

Concentration of the reactant A, that is initially dissolved in the reactor is defined (based on the final volume of reaction media) as 2 mol/liter. We accept also that the equivalent quantity of the reactant B is injected in the reactor at the moment 0 .



SINGLE-PHASE BLENDING AND REACTORS.  
INITIAL CONCENTRATIONS (LOADS) OF REACTANTS  
(BATCH & SEMIBATCH)

Init. concentration of reactant A:  mol/liter

Relation of loads - B[mol]/A[mol]:

OK Cancel Print Help

Figure 19. Entering concentration of reactant.

After the data for the pilot reactor are entered, a graph containing the results of modeling appears (Figure 20). Now the identical data are entered in the input tables of the R\_PRODUCTION project, and the corresponding output graph for this reactor is released (Figure 21).

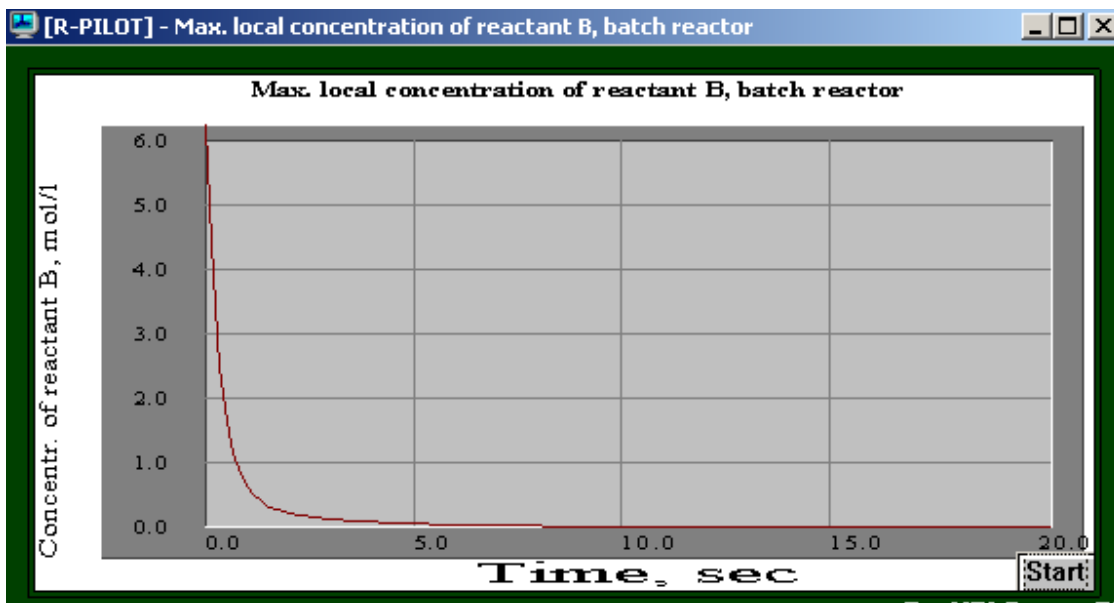


Figure 20. Pilot reactor. Distribution of the reactant B.

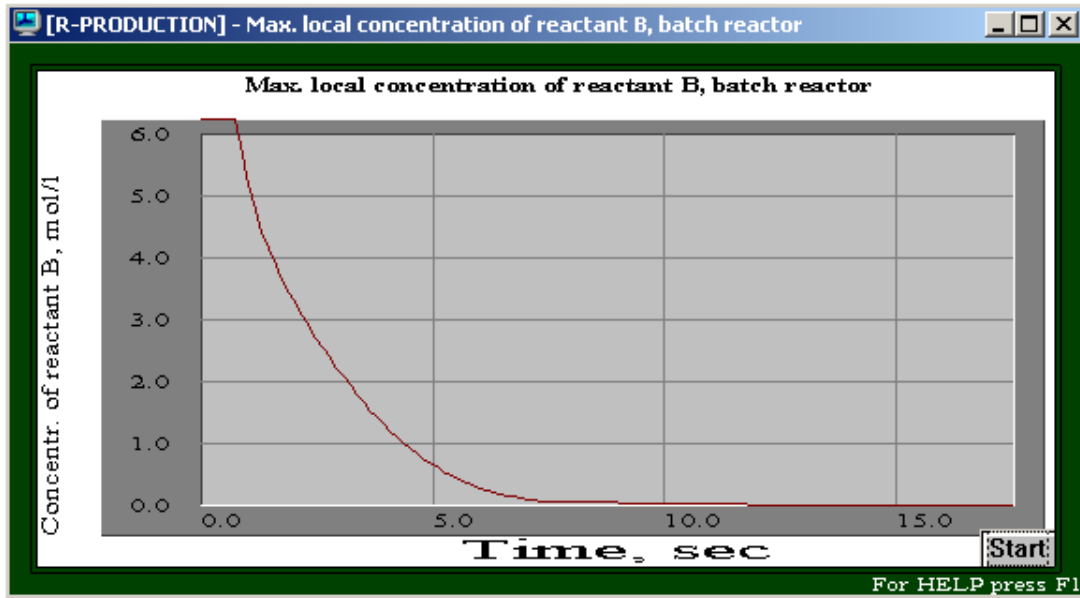


Figure 21. Production reactor. Distribution of the reactant B.

Comparing these graphs, it is easy to see that a high local concentration of the reactant B persists in the production reactor for much longer time than in the pilot reactor. For a more demonstrative comparison, we can put the graph together via **Window>Tile** (Figure 22),

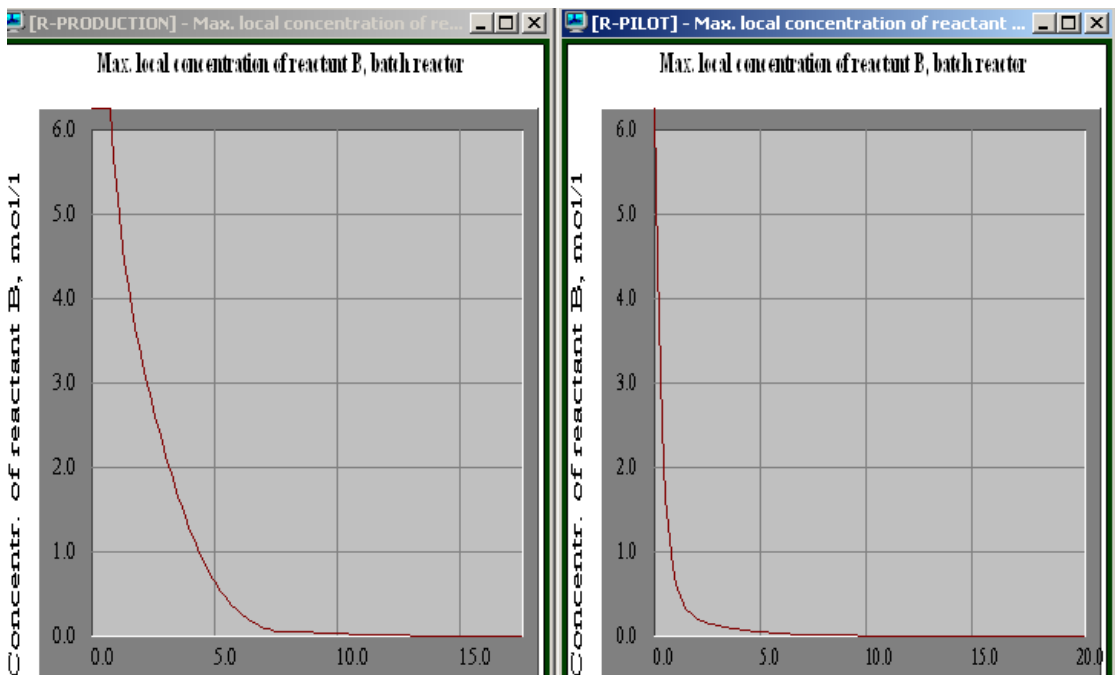


Figure 22. Comparison of the reactors for the 'FAST REACTION' approximation.

Accordingly to these results, duration of existence of a relatively high local B concentration in the production reactor is 2-3 times longer than in the pilot reactor, and it can cause the observed increase of side reaction rate.

An approximate quantitative evaluation is possible if we accept some ‘probable’ values of the kinetic constants. For example, let us enter the values presented in the Figure 23 and perform modeling of the both reactors.

SINGLE-PHASE BLENDING AND REACTORS. HOMOGENEOUS CHEMICAL REACTION	
<b>MAIN REACTION</b> $A + B = C$ Reactant A is charged initially into the tank	<b>SIDE REACTION</b> Side reaction is assumed to be slow compared to the main reaction
Specific reaction rate for BLENDING - enter 0 (zero) for FAST reaction - enter F	Reaction type $B + B = D$
1 / (mol*sec)	Specific reaction rate 0.02 / (mol*sec)
<input type="button" value="OK"/>	<input type="button" value="Cancel"/>
<input type="button" value="Print"/>	<input type="button" value="Help"/>

Figure 23. Assumed kinetic constants for comparison of the reactors.

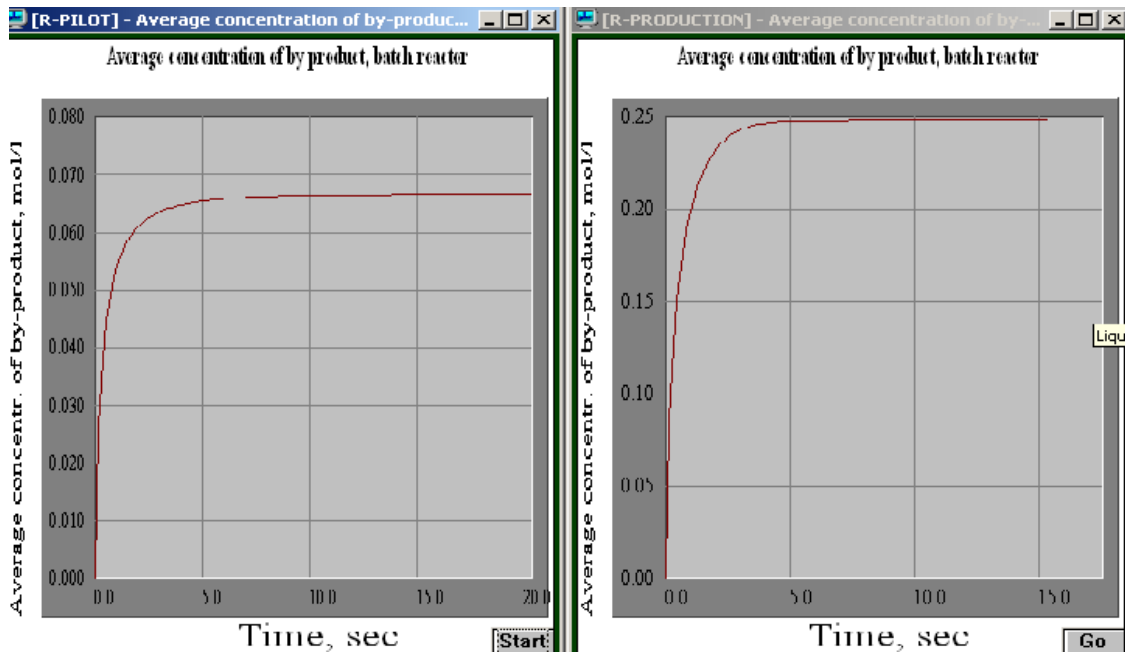


Figure 24. Comparison of Average concentration of by-product in the two reactors.

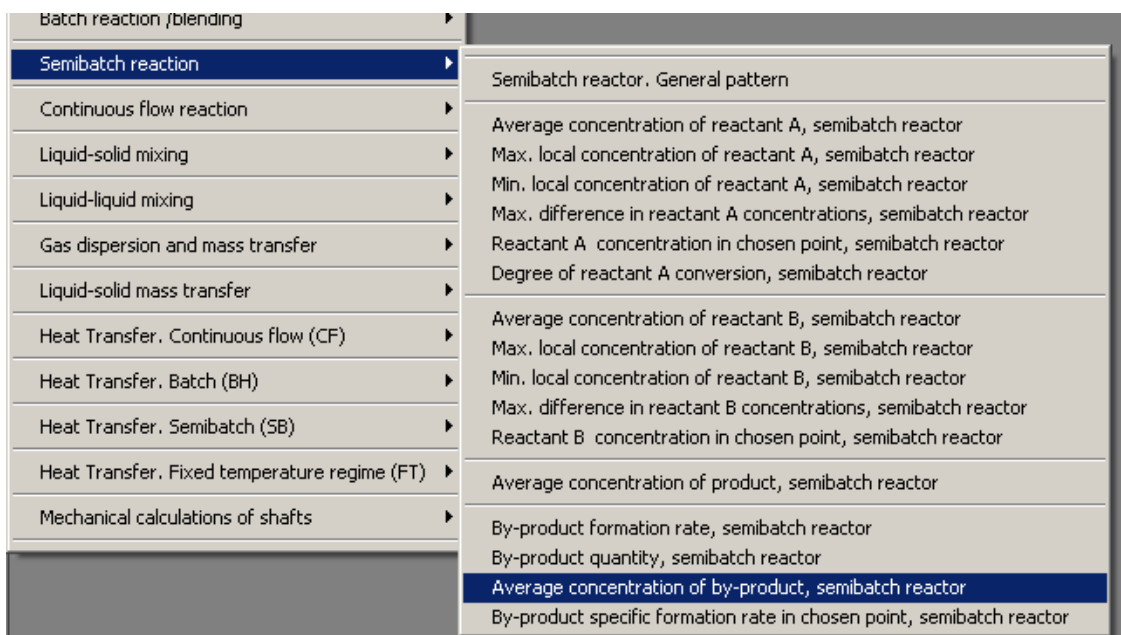
The results of calculation of the by-product concentration presented in the Figure 24 are obvious enough and can explain the unsuccessful scaling-up.

## Improvement of synthesis in the 1200 liter reactor.

**It is possible to assume that reduction of the side reaction rate and improvement of the product quality can be achieved by reduction of concentration of the reactant B in the reactor.**

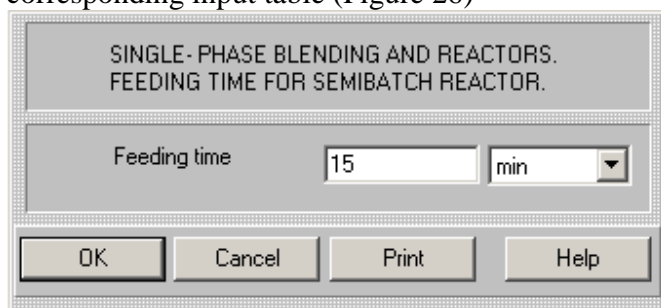
In order to achieve it without change in the reactor design, we can try to replace the batch process with a semi-batch one, i.e. to add the reactant B by small portions during 10-15 minutes.

Let us open the **Calculate menu**, select **Semibatch reaction** and click the same parameter as before – **Average concentration of by-product** (Figure 25).



**Figure 25. Semibatch reaction menu**

The program will ask us to define the feeding time for the reactant B and present the corresponding input table (Figure 26)



**Figure 26. Entering feeding time for semibatch process.**



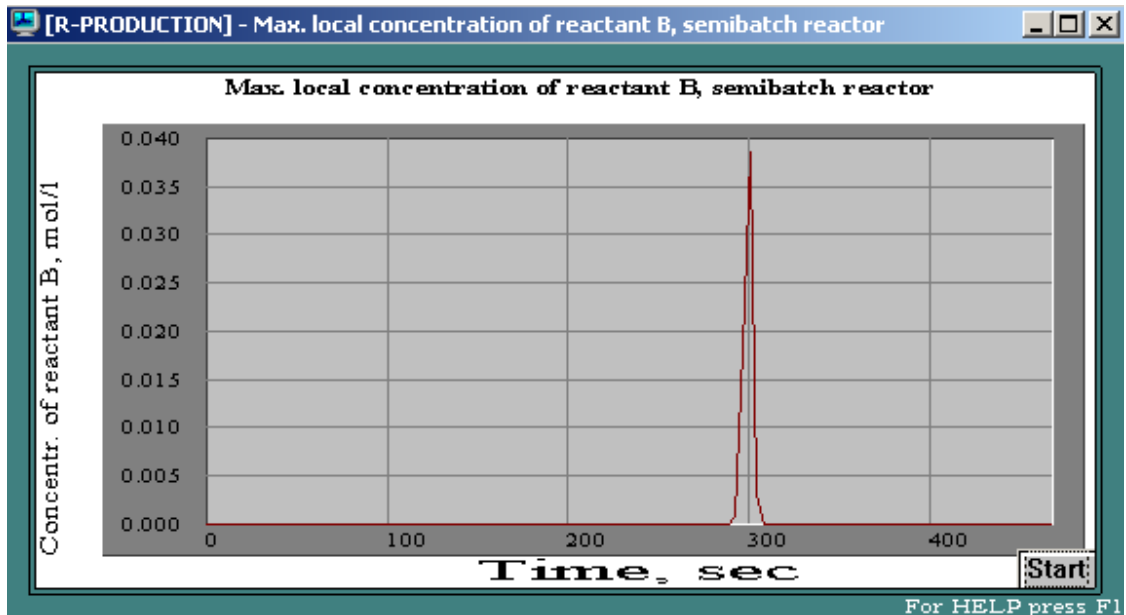


Figure 27. Max. local concentration of reactant B. FAST REACTION approximation.

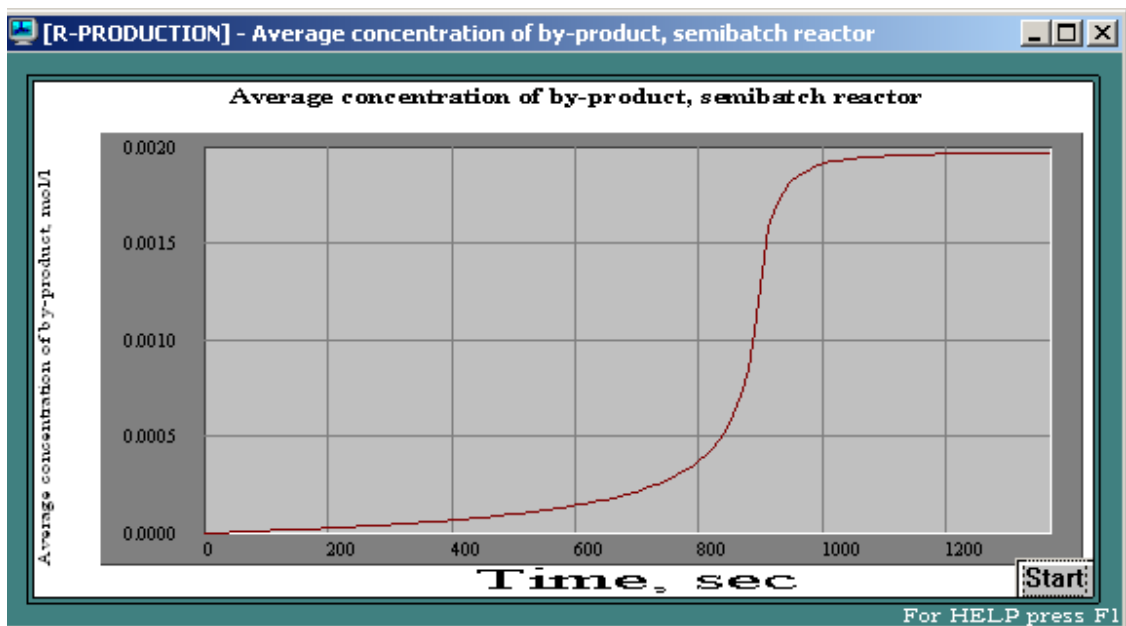


Figure 28. By-product formation in semibatch reactor (see Figure 23).

As it follows from the results of modeling presented in the Figures 27 and 28, predicted values of the max. local B concentration (for the FAST REACTION approximation) and average concentration of by-product (for kinetics defined in the Figure 23) are much lower than in the Batch reaction case.

**This solution was applied in the plant and gave positive result.**