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LOW-TEMPERATURE POLYMERIZATION CONDITIONS  
IN A FLOW-THROUGH REACTOR

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In the literature of the last few years investigations are frequently encountered devoted to the process of polymerization in a flow-through reactor. However, the amount of work in which account is taken of the dependence of the viscosity of the reaction mixture on the degree of polymerization of the substance is extremely small, although this dependence has a considerable effect on the profiles of the temperature and the degree of polymerization, the pressure drop, and other characteristics of the process. We note [1], which considered adiabatic conditions of the course of the polymerization and a weak dependence of the viscosity on the degree of polymerization was taken, [2], in which, with a number of simplifying assumptions, an analytical investigation was made of isothermal polymerization, and [3], in which an experimental investigation was made of the course of the process. The present authors have earlier investigated the process of polymerization in a flow-through continuous reactor, with a viscosity depending exponentially on the degree of polymerization  $\eta$  and the temperature  $T$ , and postulated averaging of these values over the cross section of the reactor [4, 5]. Such an approach is justified as a first approximation with calculation of the pressure drop and the distribution of the mean temperatures along the length of the reactor. However, within the framework of this approach it is impossible to establish the true distribution of the temperature, the degree of polymerization, and the velocities of the flow of the liquid over the cross section of the reactor. All these characteristics of the process are considerably affected by the dependence of the viscosity on the degree of polymerization, particularly when the polymerization takes place in the mass. With the aim of a study of the effect of the distribution of all these quantities on the course of the process, the present article considers the problem of polymerization in a tubular reactor in a two-dimensional unsteady-state statement, taking account of the dependence of the viscosity on the temperature and the degree of polymerization.

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We consider the flow of a liquid in which initiated polymerization is taking place in a finite circular tube of radius  $r_0$  and length  $L$ . It is assumed that the viscosity of the liquid depends experimentally on the temperature  $T$  and the degree of polymerization  $\eta$ :

$$\mu(\eta, T) = \mu_0 \exp(U/RT + A\eta),$$

where  $\mu$  is the viscosity coefficient;  $U$ ,  $\mu_0$ ,  $A$  are constants. Since for liquids the Prandtl number  $Pr \gg 1$ , the time of hydrodynamic relaxation is far less than the time of thermal relaxation and, from a hydrodynamic point of view, the process takes place under quasisteady-state conditions. The velocity field rapidly adjusts itself to a change in the temperature and concentration fields [6]. And since, under low-temperature conditions, the temperature and the depth of the polymerization and the associated profile of the velocities vary smoothly, then, in the hydrodynamic equations, the inertial terms can be neglected. We shall assume that the change in the axial component of the velocity  $v_x$  along the radius is considerably stronger than in the direction of the axis, and that the axial component of the velocity of the flow is much greater than the radial  $v_x \gg v_r$ . It is postulated that the heat transfer through the wall of the reactor takes place according to the Newton law, and that the heat evolution from the dissipation of energy and from the decomposition of the initiator is negligibly small. Taking account of the assumptions made, the system of equations describing the process and the initial and boundary conditions for it can be written in the form

$$\begin{aligned} \frac{dP}{dx} &= \frac{1}{r} \frac{\partial}{\partial r} \left[ \mu(T, M) r \frac{\partial v_x}{\partial r} \right]; & (1) \\ \frac{1}{r} \frac{\partial}{\partial r} (rv_r) + \frac{\partial v_x}{\partial x} &= 0, \\ \frac{\partial T}{\partial t} + v_r \frac{\partial T}{\partial r} + v_x \frac{\partial T}{\partial x} &= \frac{\lambda}{c\rho} \left( \frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial x^2} \right) + \frac{Q_0 k_0}{c\rho} e^{-E/RT} M^m I^n, \\ \frac{\partial M}{\partial t} + v_r \frac{\partial M}{\partial r} + v_x \frac{\partial M}{\partial x} &= -k_0 e^{-E/RT} M^m I^n, \\ \frac{\partial I}{\partial t} + v_r \frac{\partial I}{\partial r} + v_x \frac{\partial I}{\partial x} &= -k_{i0} e^{-E_i/RT} I^s, & (2) \\ T = T_1, M = M_1, I = I_1 & \text{ with } t = 0, \\ T = T_1, M = M_1, I = I_1 & \text{ with } x = 0, \\ \partial T / \partial x = 0 & \text{ with } x = L, \\ \partial v_x / \partial r = 0, \partial T / \partial r = 0 & \text{ with } r = 0, \\ v_x = 0, v_r = 0, \lambda \partial T / \partial x = -\alpha(T - T_2) & \text{ with } r = r_0, \end{aligned}$$

where  $r$ ,  $x$  are the instantaneous coordinates in the radial and axial directions;  $t$  is the time;  $M$ ,  $M_1$ ,  $I$ ,  $I_1$  are the instantaneous and initial concentrations of the monomer and the initiator, respectively;  $T$ ,  $T_1$ ,  $T_2$  are the temperatures of the instantaneous, initial, and surrounding medium;  $R$  is the universal gas constant;  $v_r$ ,  $v_x$  are the radial and axial components of the velocity;  $P$  is the pressure;  $\alpha$  is the heat-transfer coefficient;  $E$ ,  $Q_0$ ,  $k_0$  are the activation energy, the heat effect, and the preexponent of the polymerization reaction;  $c$ ,  $\rho$ ,  $\lambda$  are the heat capacity, the density, and the coefficient of thermal conductivity of the substance;  $E$ ,  $k_{i0}$  are the activation energy and the preexponent of the decomposition reaction of the initiator;  $m$ ,  $n$ ,  $s$  are the orders of the reactions.

Taking into consideration that the expression for the mass flow rate  $Q$  has the form

$$Q = 2\pi \int_0^{r_0} rv_r dr,$$

and integrating Eqs. (1), (2) with the appropriate boundary conditions, we can obtain expressions for the axial and radial components of the velocities and for the pressure drop. Going over to dimensionless variables we obtain

$$\begin{aligned} \frac{\partial \Theta}{\partial \tau} + q \left( v_\xi \frac{\partial \Theta}{\partial \xi} + \varepsilon v_\zeta \frac{\partial \Theta}{\partial \zeta} \right) &= \frac{1}{\delta} \left( \frac{\partial^2 \Theta}{\partial \xi^2} + \frac{1}{\xi} \frac{\partial \Theta}{\partial \xi} + \varepsilon^2 \frac{\partial^2 \Theta}{\partial \zeta^2} \right) + e^{\Theta/1+\beta\Theta} (1-\eta)^{m_i n}, \\ \frac{\partial \eta}{\partial \tau} + q \left( v_\xi \frac{\partial \eta}{\partial \xi} + \varepsilon v_\zeta \frac{\partial \eta}{\partial \zeta} \right) &= \gamma e^{\Theta/1+\beta\Theta} (1-\eta)^{m_i n}, \\ \frac{\partial i}{\partial \tau} + q \left( v_\xi \frac{\partial i}{\partial \xi} + \varepsilon v_\zeta \frac{\partial i}{\partial \zeta} \right) &= -\nu e^{\Theta/1+\beta\Theta} i^s, \\ \Delta p &= \frac{q}{\varepsilon} \int_0^1 \left( \int_0^1 \frac{\xi^2 d\xi}{\mu(\Theta, \eta)} \right)^{-1} d\zeta, \quad \mu(\Theta, \eta) = \exp \left( A\eta - \frac{\sigma\Theta}{1+\beta\Theta} \right), \end{aligned}$$

$$v_{\xi} = -\frac{\varepsilon}{2} \frac{\partial}{\partial \zeta} \left[ \left( \int_{\xi}^1 \frac{\xi d\xi}{\mu(\Theta, \eta)} + \frac{1}{\xi} \int_0^{\xi} \frac{\xi^2 d\xi}{\mu(\Theta, \eta)} \right) \right] \bigg/ \int_0^1 \frac{\xi^2 d\xi}{\mu(\Theta, \eta)}, \quad v_{\zeta} = \int_{\xi}^1 \frac{\xi d\xi}{\mu(\Theta, \eta)} \bigg/ \int_0^1 \frac{\xi d\xi}{\mu(\Theta, \eta)}.$$

The initial and boundary conditions:  $\Theta = \Theta_1$ ,  $\eta = 0$ ,  $i = 1$  with  $\tau = 0$ ,  $\Theta = \Theta_1$ ,  $\eta = 0$ ,  $i = 1$  with  $\zeta = 0$ ,  $\partial\Theta/\partial\xi = 0$  with  $\zeta = 1$ ,  $\partial\Theta/\partial\xi = 0$  with  $\xi = 0$ ,  $\partial\Theta/\partial\xi = -\text{Bi}(\Theta - \Theta_2)$  with  $\xi = 1$ .

Here the following notation is introduced:

$$\begin{aligned} \xi &= \frac{r}{r_0}, \quad \zeta = \frac{x}{L}, \quad \tau = t \frac{EQ_0 k_0 M_1^m I_1^n}{c\rho RT_0^2} \exp\left(-\frac{E}{RT_0}\right), \quad v_{\xi} = \frac{v_r}{v_0}, \\ v &= \frac{v_x}{v_0}, \quad \Theta = \frac{E}{RT_0^2}(T - T_0), \quad \eta = \frac{M_1 - M}{M_1}, \quad i = \frac{I}{I_1}, \\ \mu(\Theta, \eta) &= \frac{\mu(T, M)}{\mu(T_0, M_1)}, \quad p = P \frac{c\rho RT_0^2}{2\mu(T_0, M_1) M_1^m I_1^n} \exp\frac{E}{RT_0}, \quad \varepsilon = \frac{r_0}{L}, \\ q &= \frac{Qc\rho RT_0^2}{\pi r_0^3 M_1^m I_1^n k_0 EQ_0} \exp\frac{E}{RT_0}, \quad \beta = \frac{RT_0}{E}, \quad \text{Bi} = \frac{\alpha r_0}{\lambda}, \quad \omega = \frac{E_i}{E}, \quad \sigma = \frac{U}{E_i}, \\ v_0 &= \frac{Q}{\pi r_0^2}, \quad \delta = \frac{r_0^2 Q_0 E k_0 M_1^m I_1^n}{\lambda RT_0^2} \exp\left(-\frac{E}{RT_0}\right), \quad \gamma = \frac{c\rho RT_0^2}{M_1 EQ_0}, \\ v &= \frac{k_{i0} I_1^{s-n-1} c\rho RT_0^2}{k_0 M_1^m EQ_0} \exp\frac{E - E_i}{RT_0}, \quad \Theta_1 = \frac{E}{RT_0^2}(T_1 - T_2), \\ \Theta_2 &= \frac{R}{RT_0^2}(T_2 - T_0). \end{aligned}$$

The most important parameters of the problem are:  $q$  is the dimensionless mass flow rate;  $\Theta_1$  is the dimensionless initial pressure drop;  $\delta$  is the Frank-Kamenetskii parameter, characterizing the ratio of the time of the arrival of heat from the chemical reaction to the time of the heat evolution.

The problem was solved numerically on a computer with the following fixed values of the parameters:  $A = 8$ ,  $\sigma = 0.2$ ,  $\beta = 0.036$ ,  $\omega = 1.38$ ,  $\gamma = 0.032$ ,  $\nu = 0.0013$ ,  $\varepsilon = 0.002$ ,  $\text{Bi} = 1.4$ ,  $m = 1$ ,  $n = 0.5$ ,  $s = 1$ . The chosen scale of the temperature  $T_0$  was the temperature of the surrounding medium  $T_2$  ( $\Theta_2 = 0$ ). It was shown in [3] that the solution depends only weakly on the value of the parameters  $\beta$ ,  $\gamma$ ,  $\nu$ ,  $\omega$ , and that the process can take place under two sets of sharply differing conditions: low-temperature and high-temperature. Under critical conditions there exists a functional dependence between the parameters of the problem. With the above values of the parameters and two values of  $\Theta_1 = 3.5$  and  $-3.5$ , there are respectively obtained the following critical values of the parameter  $\delta$ :  $\delta_* = 0.11 \pm 0.01$  and  $0.035 \pm 0.005$ . We note that, under low-temperature conditions, the values of the parameters  $\text{Bi}$  and  $\delta$  have only a slight effect on the course of the process, as a result of the small degrees of heating of the substance.

Let us examine the course of the polymerization process with  $\Theta_1 < 0$ , i.e., when the cold substance flows into a hot reactor. It was assumed that at the initial moment of time  $\tau = 0$  the reactor is filled with a cold mixture of monomer and initiator ( $\eta = 0$ ,  $i = 1$ ,  $\Theta = \Theta_1$ ). The same mixture at the same temperature  $\Theta_1$  is supplied continuously to the reactor, and the reaction products are removed from the reactor. The rate of feed of fresh substance and of the removal of the reaction products is determined by the mass flow rate  $q$ , which is maintained constant. Heating of the liquid occurs both due to heat transfer with the surrounding medium through the walls of the reactor and due to heat evolution during the course of the polymerization reaction.

At the initial temperature the rate of the reaction is not great but, with an increase in the temperature, due to heating of the substance the rate rises. However, due to burn-up of the substance, the increase in the reaction rate is slowed and, in the final analysis, the system arrives at some steady state. During the period of the establishment of a steady state, depending on the conditions of the problem, the curve of the dependence of the maximal temperature on the time can be monotonically rising or can have a maximum. The steady-state profiles of the temperature are given below, along with a discussion of them.

The form of the steady-state temperature field, with other parameters being given, depends on the value of the mass flow rate  $q$ . We note that, with relatively small mass flow rates, a chemical source of heat heats the reaction mixture above the temperature of the walls of the reactor. Starting from some mass flow rate, the temperature of the substance is always less than the temperature of the walls, since the residence time of the substance in the reactor becomes less than the heating time, and the substance, not having an opportunity to be heated up, flows out of the reactor cold ( $\Theta < 0$ ).

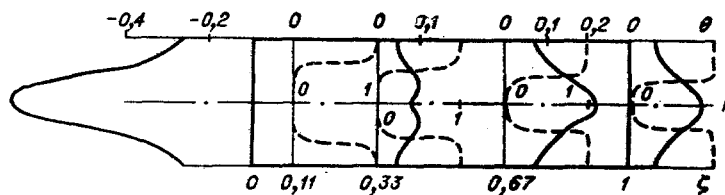


Fig. 1

The formation of a steady-state distribution of the temperature and, in particular, of the degree of polymerization, is considerably affected by the distribution of the axial component of the velocity of the flow. As a result of the adhesion of the liquid to the walls and to the presence of a distribution of the velocity over the radius, the prewall layer of liquid remains in the reactor for a considerably longer time than the liquid near the axis of the reactor. Moreover, the prewall layers are heated more rapidly. Therefore, the substance in them reacts to a greater depth. In the case of a strong dependence of the viscosity on the degree of polymerization this leads to stagnation of the prewall layers, which promotes a further increase in the degree of polymerization. Due to the stagnation of the liquid at the walls there is an increase in the velocity of the flow of the liquid in the preaxial part of the reactor. As a result, there is a deterioration of the conditions of heat removal to the preaxial region and the residence time of these layers in the reactor is reduced.

Figure 1 illustrates with dashed lines the curves of the distribution of the degree of polymerization  $\eta$  over the reactor in different cross sections with  $q = 20$ ; it can be seen that, near the wall, as a result of the stagnation of the liquid and the more favorable temperature and time conditions, there is total conversion of the substance ( $\eta \approx 1$ ). Due to the sharp rise in the viscosity with an increase in the degree of polymerization, there is adhesion of the reacted mass to the walls of the reactor and a constriction of its live cross section. Since the mass flow rate of the liquid is constant, the formation of a very viscous, almost immovable layer at the wall of the reactor leads to an acceleration of the flow of the liquid near the axis of the reactor. A characteristic jet of low-viscosity liquid with a small degree of polymerization  $\eta$  is formed. In Fig. 1 there is a sharp delineation of the adhering layer at the wall with  $\eta \approx 1$  and the low-viscosity jet of liquid, in which the degree of polymerization does not exceed a few percent. The solid lines show the steady-state profiles of the temperature of the substance in the reactor with the same flow rate  $q = 20$ . It can be seen that, with the motion of the substance along the reactor, the liquid is at first heated almost like a solid body; the chemical source of heat then makes a considerable contribution. Since the prewall layers are heated more rapidly, the chemical sources of heat coming into play promote the formation of a maximum of the temperature in these layers. Subsequently, with burn-up of the substance, the chemical sources in these layers cease to act, and the maximum of the temperature is shifted toward the axis of the reactor.

Figure 2 illustrates the profiles of the axial velocities of the liquid with different values of the mass flow rate. With small mass flow rates the velocity of the flow of the liquid, even at the axis of the reactor, is so small that the monomer can react completely. There is filling of the whole cross section by the polymer mass. The flow of the low-viscosity liquid in the reactor takes place in the form of an extended jet in the thickness of the polymer mass. Figure 2a illustrates this case. The dashed lines show the boundary between the adhering layer and the low-velocity liquid. Beyond the limits of this zone, the viscosity of the liquid is approximately constant over the cross section (a constant degree of polymerization,  $\eta \approx 1$ ), and the profile of the axial velocities is close to parabolic.

With an increase in the mass flow rate, the steady-state profiles of  $\theta$ ,  $\eta$ , and  $v_z$  change. The residence time of the substance in the reactor is reduced, the jet is extended, and, at a given value of the mass flow rate, reaches the outlet from the reactor. In this case, the motion of the substance in the reactor takes place mainly in a narrow jet of low-viscosity, but almost unreacted, liquid. A large part of the reactor is filled by the polymer mass, which is displaced very slowly. With a further rise in the mass flow rate (Fig. 2b, c), as a result of a decrease in the residence time of the substance in the reactor, the rate of polymerization of the low-viscosity substance in the jet falls. With an increase in the mass flow rate, there is an increase of the shear stress in the jet of low-viscosity liquid, which leads to an increase in the rate of motion of the liquid in the transitional region from the jet to the adhering layer. As a result, the residence time of the liquid in the reactor in this transitional zone is shortened, which leads to an expansion of the jet with a rise in the mass flow rate. With an expansion of the jet (an increase in the live cross section of the reactor) the relative velocity of the flow in the jet decreases.

With very large mass flow rates, the liquid leaves the reactor with no opportunity to be heated or to react, with the exception of a thin layer near the wall; in this case the profiles of the axial velocities are close to parabolic.

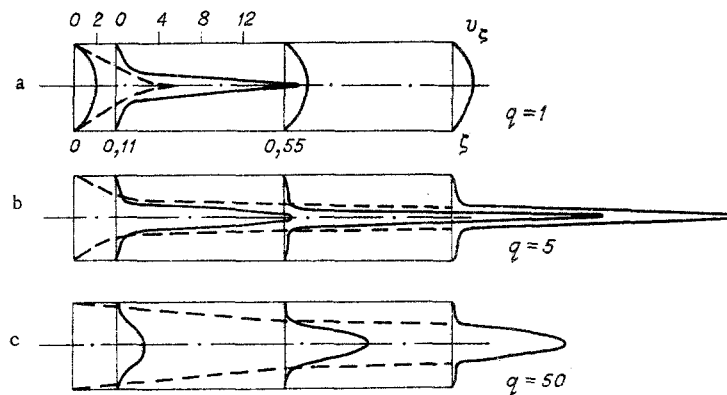


Fig. 2

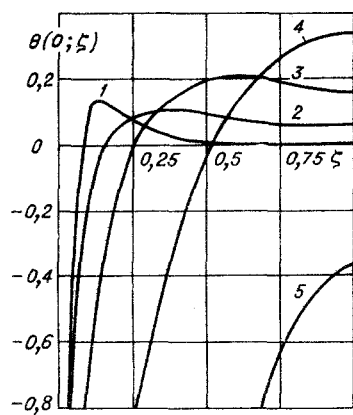


Fig. 3

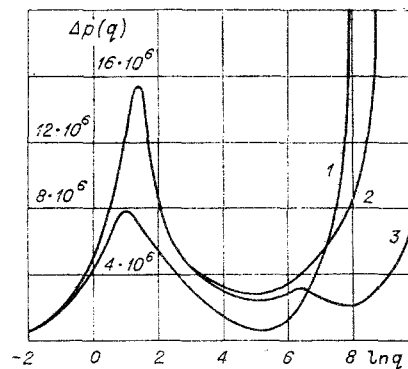


Fig. 4

Figure 3 shows the change in the steady-state distribution of the temperature at the axis of the reactor with a change in the mass flow rate. The curves were calculated with the following values of the mass flow rate: 1)  $q=1$ ; 2) 5; 3) 20; 4) 100; 5) 300. In the case of very small mass flow rates, the whole curve lies below the axis of abscissas, by virtue of the smallness of the amount of heat entering the system from the reaction. With a rise in the mass flow rate, the amount of heat entering the system due to the chemical reaction rises and, in a certain range of change of the mass flow rate, there is heating of the liquid. Here, depending on the value of the mass flow rate, the curve can have a maximum or can be monotonically rising. Out of all the curves, the maximum is most sharply marked for the first curve, corresponding to the case where the low-viscosity jet lies wholly within the reactor. The reaction practically goes to completion in the region occupied by this jet; the liquid heated during the course of the reaction is then cooled and the temperature of the liquid tends toward the temperature of the surrounding medium. With an increase in the mass flow rate, the jet is extended and the maximum of the curve is shifted toward the outlet from the reactor; here, the value of the maximum at first decreases somewhat (curve 2) and then rises (curves 3, 4). The smallest value of the maximal temperature corresponds to a mass flow rate with which the jet reaches the end of the reactor. The decrease in the maximal temperature is connected with an extension of the region in which there is heat evolution. After "breakdown" the extension of the jet ceases, and, with a rise in the mass flow rate, there is an increase in the region occupied by jets. This leads to a rise in the maximal temperature until a second factor comes into play, connected with the convective entrainment of unreacted liquid. Due to the action of this factor, with a further increase in the mass flow rate, the maximal temperature at the axis is lowered and, with large mass flow rates, becomes negative, i.e., the liquid in the volume cannot be heated up to the temperature of the surrounding medium (curve 5).

Of great interest with polymerization in a flow-through reactor is the pressure-mass-flow characteristic of the reactor, curve  $\Delta p(q)$ . Figure 4 shows in semilogarithmic coordinates the pressure-flow-rate characteristics for the cases of negative and positive inlet temperatures of the substance. The curves were calculated with the following values of the parameters: 1)  $\Theta_1 = -3.5$ ,  $\sigma = 0.2$ ; 2)  $\Theta_1 = 3.5$ ,  $\sigma = 0.2$ ; 3)  $\Theta_1 = 3.5$ ,  $\sigma = 0.8$ . Curve 1 has a maximum and a minimum. If the liquid flows in a tube, with a constant viscosity, the pressure drop rises in direct proportion to the rise in the mass flow rate of the liquid. In the case under consider-

eration, even with small flow rates, the curve of  $\Delta p(q)$  deviates from a linear dependence, corresponding to the case of the Poiseuille flow of a completely reacting liquid having a temperature  $\Theta = 0$ . This deviation takes place due to the entrainment of a very viscous substance with  $\eta \approx 1$  with a rise in the mass flow rate, and to an increase in the fraction of the low-viscosity substance in the reactor. With the growth of the jet this deviation increases. The maximum of the curve of  $\Delta p(q)$  corresponds to a mass flow rate such that the jet reaches the outlet from the reactor and there is a "breakdown" of the viscous mass by the low-viscosity liquid. With a further increase in the mass flow rate, the pressure drop decreases as a result of the expansion of the jet of low-viscosity liquid and a sharp increase in the fraction of unreacted liquid in the reactor. An increase in the mass flow rate leads, finally, to entrainment of the heated substance and to filling of the whole reactor with cold liquid. As has already been remarked, in this case, at the wall of the reactor there exists a thin viscous layer, and practically the whole cross section of the reactor is filled with cold unreacted liquid ( $\eta < 1\%$ ). Therefore, after the minimum of the pressure-mass-flow curve for a mass flow rate with which the steady-state temperature of the substance in the reactor becomes negative over the whole volume, the pressure drop rises approximately in proportion to the mass flow rate.

Curves 2, 3 show the pressure-flow-rate characteristics with a positive value of  $\Theta_1 = 3.5$ , i.e., where a hot substance is fed into a cold tube (such a situation can occur in processes of the additional polymerization of a substance after it leaves the reactor). These curves differ in the value of the parameter  $\sigma$ , i.e., the degree of dependence of the viscosity on the temperature. A strong dependence of the viscosity on the temperature (or a large value of the initial temperature drop  $\Theta_1$ ) brings about a second drop in the pressure-flow-rate characteristic in the region of large mass flow rates, where the distribution of the velocities of the flow over the cross section of the reactor is close to parabolic. Curve 3 has two maxima. The secondary decrease in the pressure drop with a rise in the mass flow rate takes place as a result of the entrainment of colder and, consequently, more viscous liquid from the tube and the filling of the tube with hotter and less viscous liquid. With mass flow rates such that the temperature drop and the change in the viscosity along the length of the tube become insignificant, the pressure drop again rises by virtue of the linear dependence on the mass flow rate. A similar behavior of the curve of  $\Delta p(q)$  for the flow of an inert liquid was investigated in detail in [7]. It is interesting that, in the case under consideration, in a determined range of change in the parameters, the regions of a significant effect of factors of the dependences of the viscosity on the temperature and the degree of polymerization on the pressure-flow-rate characteristics are distinct, and each of these factors generates its own maximum on the curve of  $\Delta p(q)$ .

We note that the curve of  $\Delta p(q)$  does not differ qualitatively from the pressure-flow-rate characteristics obtained in [4, 5] without taking the two-dimensional nature of the process into consideration.

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