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POLYMERIZATION IN THE PROCESS OF VISKOZIMETRIK MEASUREMENTS OF TECHNOLOGIES FOR IMPROVING QUALITY CONTROL RESEARCH PROBLEMS

Specialty: 3337.01 Information - measuring and control system
(in the chemical industry)

Field of science: Technical sciences

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ABSTRACT

on the presented dissertation to get an academic degree of philosophy doctor of technical science

SUMGAIT-2022

The dissertation work was performed at Sumgait State University, Information and Computer technology department.

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GENERAL CHARACTERISTICS OF WORK

Relevance of the topic and degree of development .

The fact that the problem of controlled quality is one of the central topics in modern polymer materials technology requires the creation of measurement tools or the improvement of existing systems in terms of many physical measurements. In the course of the polymerization process, a number of quality indicators of the reaction medium, which are encountered as a necessity, either do not exist or the existing measurement principles do not meet the requirements, create serious problems in terms of quality control.

Polymerization in the mass, which is a periodic process, is remarkable in this respect again because the existence of an after-effect in the dynamics of polymerization brings the concept of regular measurement to the fore. Quality control, and especially its management, presents the approach to the polymerization reactor from the dynamic system position and, accordingly, the understanding of the system's state vector and optimal trajectory as a research issue.

The reaction medium inside the polymerization reactor is a source of viscometric information rich enough to reflect the course of the process, having the most typical properties representing non-Newtonian fluidity. It is shown by irrefutable facts in the scientific and technical literature of recent years that the acquisition, processing and application of this information to the management process has an exceptional role in solving the issue of quality management.

These issues have not been discussed in the scientific and technical literature until today. In such conditions, identification of the current state vector of the polymerization process and its use as operational information in management is one of the urgent issues of high-quality polymer production.

Object and subject of the research: The considered object is a polymerization process operating mainly in periodic production

mode, consisting of a non-Newtonian high-molecular liquid environment characterized by the change of its parameters over time. The subject of research is a polymer mass with given quality indicators It consists in examining the issue of determining optimal management effects as a guarantee of achievement.

Aims and objectives of the research: The aim of the research is to improve the principles of determining the current molecular mass distribution functions based on rheological measurements, which most comprehensively reflect the dynamics of monomer conversion in periodic polymerization processes. As a result of the study of the rheological research of the reaction medium in this field in relation to various technological conditions in terms of informatics, the design of an appropriate measuring device is directly proposed as the goal of the dissertation work.

Research methods. In the research, the method of parametrization of statistical analysis with n-order moments, rotational analysis method in the field of fluid mechanics, computer modeling and optimal control methods were used.

The main provisions defended :

1. Proof that the fluidity characteristic of the reaction medium in the polymerization process is a sufficiently adequate indicator of the dynamics of the formation of the quality indicators of the product;

2. High informativeness of viscometric analysis in terms of quality control by variable speed rotation method;

3. The method of using the parameters of electric transmission in measuring the viscous resistance moment in the rotation system with relief cylindrical surfaces;

4. Concept of state space of the reaction environment in the issue of polymerization process management;

5. The issue of automatic control of the gel-effect;

6. The issue of approaching the given quality indicator in the polymerization process.

Scientific innovations of research. The scientific innovations of the dissertation consist of the following:

1. The working principle of variable speed rotational viscometer with relief cylindrical surfaces and the development of the method of measuring viscous resistance moment.

2. Finding that the analysis of the given class of flow functions into the space of three initial moments has the property of statistical separation on the basis of the calculation experiment methodology.

3. Formulation and solution of quality control problem based on variable speed rotational viscometry.

4. Formulation of the problem of determining the linear operator, which maps the vector of parameters of the flow function to the space of initial moments of the molecular-mass distribution function, and the algorithm of the solution.

5. Mathematical processing and solution algorithm of the problem of early prediction of the gel effect in the periodic polymerization process.

6. Mathematical formulation and solution method of the desired quality problem by introducing the concepts of polymerization process state space and optimal trajectory into the quality control problem.

Theoretical and practical significance of research.

By parametrizing the molecular mass distribution functions on the basis of t-order initial moments, the algorithm of their injection into the space of quality parameters of polymer masses can be used as an efficient method in the technology of polymer production and also in the technology of a number of high molecular compounds. The obtained theoretical and practical problem-solving algorithms can be applied in operational management systems of technological processes of polymer materials production.

Approval and application. The main provisions of the dissertation were discussed at the meetings of the Scientific Council of Sumgayit State University, as well as at the following scientific and technical conferences and forums: Sumgayit, II Republican scientific conference, Sumgayit, 2012; Materials of the international scientific conference on modern scientific-technical and application

problems of energy, Sumgayit, 2015; Republican scientific conference dedicated to Academician Togrul Shakhtakhtinsky's 90th anniversary, Baku, 2015; Materials of the International Scientific and Technical Conference dedicated to the Day of the Chemist and the 40th anniversary of the Department of Chemical and Technological Processes of the Branch of the Ufa State Petroleum Technical University in г. Salavate, UFA, 2017; Materials of the XXI Republican scientific conference of doctoral students and young researchers, Baku, 2017; Information systems and technologies achievements and perspectives. Proceedings of the international scientific conference, Sumgayit, 2018; Information systems and technologies achievements and perspectives Proceedings of the international scientific conference, Sumgayit, 2020.

The name of the institution where the dissertation work was performed. Dissertation work was performed at the Department of Process Automation of Sumgayit State University.

The total volume of the dissertation is indicated by noting the volume of the structural sections of the dissertation separately . The dissertation was written in accordance with the requirements set by the Higher Attestation Commission under the President of the Republic of Azerbaijan. Dissertation title page 421 conventional mark number, table of contents 7245 conventional index number, introduction 5699 conventional index number, consists of four chapters, conclusion, bibliography and appendices. The main content of the work consists of 147 pages, 17 pictures, 24 graphs and 3 tables. 106 sources are listed in the bibliography. Dissertation consists of 204084 characters without tables, figures and bibliography.

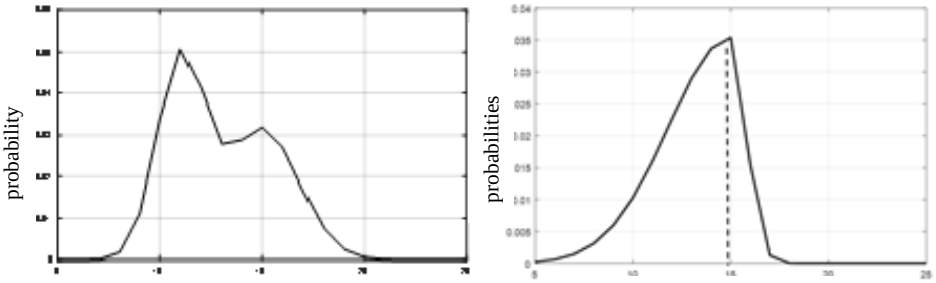
MAIN CONTENTS OF THE WORK

The **introductory** part of the dissertation, the relevance of the dissertation topic is justified, the purpose of the research is explained, the main issues that need to be solved are defined, the main propositions that are defended are indicated, and the scientific

innovations and practical significance of the obtained results are indicated.

The first chapter provides an analysis of the chemical essence of polymerization processes, the diversity of technological structures, and the principle and apparatus realizations of the necessary measurements corresponding to them. Molecular mass distribution functions (MMDF) formed in connection with the molecular-kinetic mechanism of polymerization special features are noted. By providing information about the practical measurement methods of MMDEs, in parallel, their comparative analysis and operational difficulties in production conditions are highlighted. A comparative analysis of the osmometric method and the exclusion or gel permeation chromatography methods in terms of degree of conformity and accuracy to the conditions of the polymerization reactor is given.

Here , the issue of approximation of molecular mass distribution functions of polymers by means of Gaussian function parameters is proposed. Mathematical expressions in two constructions are proposed for the approximation of empirical distribution functions by two- and three-parameter Gaussian functions in the interval of variation of the polymerization number. In one version of them , the (4+1) variation parameter included in the sum of two functions with the participation of both *Gaussian parameters, and in the other, i.e., the 4-parameter identification problem corresponding to the variant in which the asymmetric Gaussian is* taken, is required. Fig.1 . shows the variants of approximating functions based on face parameter and asymmetric Gaussian in a .



a) b)

Fig. 1. Approximation with Gaussian functions.
(a) Examples of approximations with the sum of two Gaussian functions and an asymmetric Gaussian (b).

The distribution functions sought in the 5-parameter variant of the approximation are expressed as the following sum:

$$f(x) = \frac{\mu}{\mu_0}\varphi_1(x) + (1-\mu)\varphi_2(x);$$

$$\varphi_1(x) = \frac{1}{\sigma_1\sqrt{2\pi}} e^{-(x-\bar{x}_1)^2/2\sigma_1^2}; \varphi_2(x) = \frac{1}{\sigma_2\sqrt{2\pi}} e^{-(x-\bar{x}_2)^2/2\sigma_2^2}; \mu_0 = \frac{\xi_1}{\xi_2}; \quad x \in [a,b]$$

where that are ξ_1, ξ_2 – the integral values of the $\mu, \bar{x}_1, \bar{x}_2, \sigma_1, \sigma_2$ – relevant distribution functions over the variation interval (i.e. over the interval) of their arguments , $x \in [a,b]$ and are the variational parameters.

The variation parameters in the four-parameter asymmetric Gaussian variant are expressed as follows:

$$F(x) = \frac{1}{\xi}[\frac{\sigma_2}{\sigma_1}\underset{x\in[a,\bar{x}]}{\varphi_1(x)} + \underset{x\in[\bar{x},b]}{\varphi_2(x)}]$$

where the functions $x \in [a,\bar{x}]$ involved in the approximation differ only $\varphi_1(x), \varphi_2(x)$ in the $x \in [\bar{x},b]$ determination intervals and σ_1, σ_2 diversity of variances.

Some of the proposed asymmetric Gaussian approximations are given in Fig . 2.

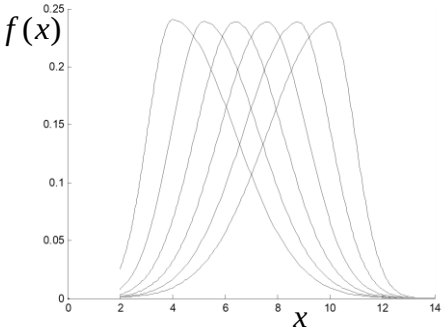


Fig. 2 . Graphical representation of a family of functions that satisfy the requirements imposed on molecular distribution functions.

In order to evaluate the accuracy of such an approximation, the reconciliation of the synthesized distribution functions with the empirical sample based on the mean square bias criterion was used:

$$F = \int_a^b \left(\tilde{f}(x) - f(\bar{x}, \sigma_1, \sigma_2) \right)^2 dx \to \min$$

where the $\tilde{f}(x)$ – empirical function $f(\bar{x}, \sigma_1, \sigma_2)$ represents the approximating function that minimizes the deviation by changing its parameters in limited intervals.

Then, Chapter 1 provides information on the analysis of the most necessary measurements from the point of view of quality control, the principles of measurement and the design features of the devices, the Muni viscometer, and the constant speed rotational rheometer. Based on this analysis, the purpose and essence of the quality assessment and management issues to be solved based on viscometric measurement and analysis are explained in chapter 1.

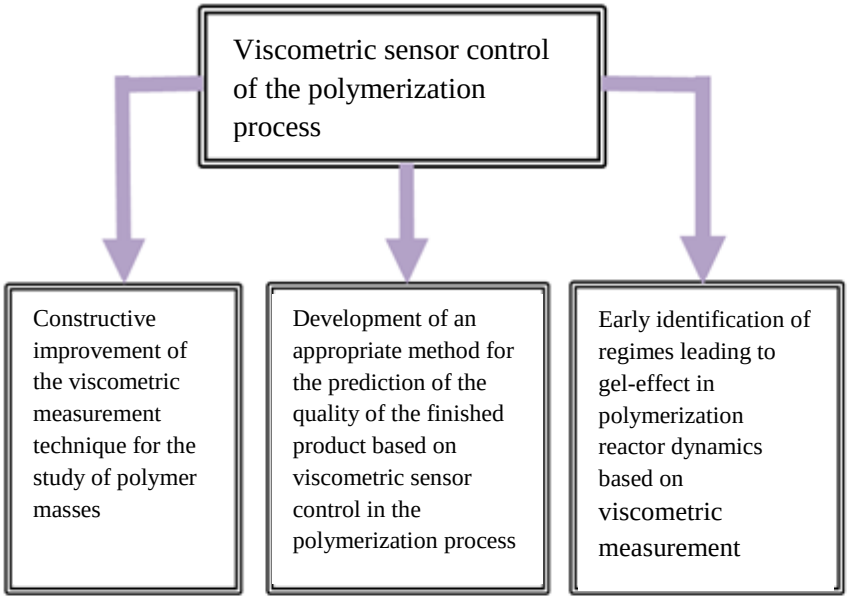


Fig. 3 . Key functional issues posed for sensor control of polymerization processes.

The second chapter is devoted to the issues of rotational rheological study of a non-Newtonian fluid medium with a polymer-based polydisperse structure. Here the internal resistance forces in the liquid medium, viscosity and non-Newtonian flow function, anomalous viscosity and its types are brought into focus. Based on the information obtained from the scientific literature, it can be concluded that many of the types of fluidity mentioned in the study of polymerization processes (and according to some data, the majority) manifest themselves (Fig. 4) .

In the picture: 1- characteristic with Newton dependence; 2- dilatant fluidity; 3- pseudoplastic fluidity; - 4-sharp nonlinear pseudoplastic flow; 5- bingam fluency is raised.

In the 2nd chapter, traditional rheological research technologies and the barriers that appear in the analysis of polydisperse fluid

media are analyzed, and a measurement method and constructive structure that serves to overcome these barriers is proposed. This principle is based on rheological analysis based on a rotary device.

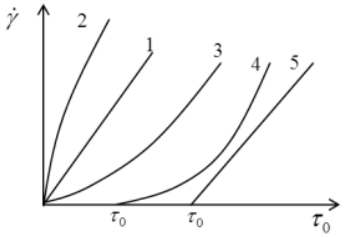


Fig . 4 Qualitative diversity of non-Newtonian flow curves.

The problem is that it is much more difficult to find two different materials with the same flow curve in constant rotation speed experiments. In other words, in experiments (measurements) with variable rotation speed, the result reveals the special properties of the material to an incomparable extent.

Fig . 5 shows the fluidity curve obtained from the experiment conducted on epoxy resin.

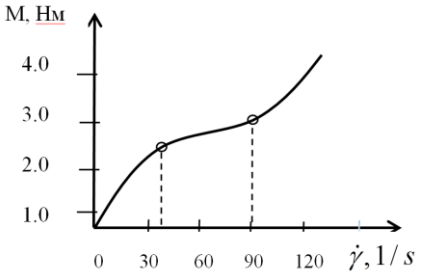


Fig . 5 . Characteristic "S"-shaped flow curve.

A variable speed viscometric device with embossed cylindrical surfaces.

It should be noted that one problem of the viscometric measurement-control system is related to the processing of fluidity curves, and the other is the creation of effective principles and devices for obtaining those curves. For this purpose, our proposal in the field of creating variable speed rotation viscometers consisted of the following. The fundamental difference of the device from similar measuring devices lies in the creation of unevenness in a certain arrangement on the surface of cylindrical structures (glasses) that slide relative to each other. The leading idea of this relief is related to the sharp response to the rotational speed of the viscous resistance that the obstacles will create in the medium between the cylinders. The degree of aggregation formed in the reaction medium and the polydispersity index have a significant effect on the fluidity function as a result. The basis of this idea comes from the fact that the moment of resistance falling on the mixers used in polymerization devices is closely correlated with the state of the reaction mass.

The principle rotation elements of the laboratory device proposed for the viscometric study of the viscous medium (cups: "rotor-sator" pair) are shown in fig. 6 is shown.

Fig. 6 . Viscometric device sensor elements;
a) stator, b) rotor.

Fig . 7 shows the laboratory device of the rotational viscometer based on the resistance moment measurement based on the proposed principle.

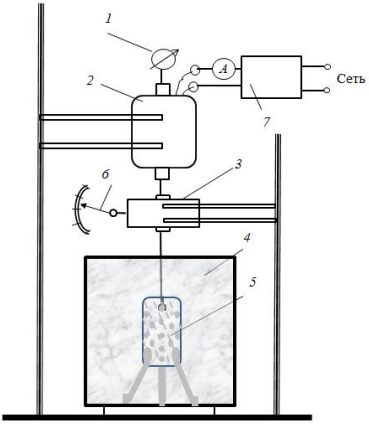


Fig . 7 . Obtaining the fluidity function of the viscous medium based on the rotational electromechanical characteristic, three laboratory devices.

The main parts of the device: 1- tachometer measuring rotation speed, 2- direct current motor, 3- stepless friction transmission (variator), 4- tank filled with the researched material, 5- sensitive element of the device (rotor-stator pair with relief). , 6- indicator dial, 7-voltage stabilizer.

The voltage stabilizer plays an important role in the device as a guarantor of measurement accuracy. The frictional contact force of the variator is tuned to minimize slippage between the wheels in stationary modes. By means of an angle indicator connected to the output shaft of the variator, the gear ratio is visually noticed and recorded.

Calibrating a measurement based on this principle requires the use of a reference material and a reference measuring device (reference device). As the initial stage of the research, in the considered work, only the flow curve was obtained for the taken test material. For this, the tank was filled with ЕД-20 brand epoxy resin

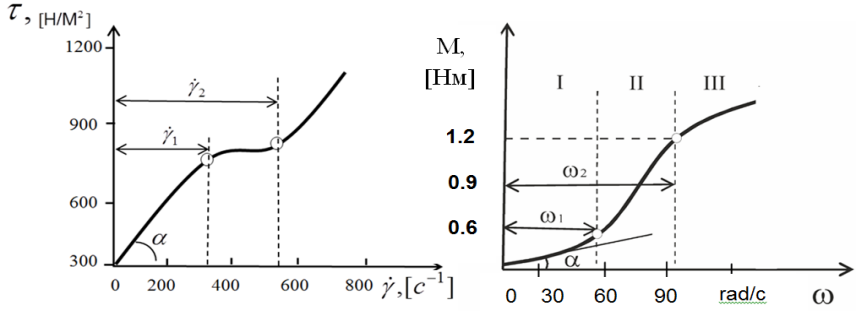
with a known viscosity coefficient (12-20 Pa/s), the rotation system was started after the gear ratio of the variator was brought to the minimum value, and the speed was gradually increased by 5-10% every 10-15 seconds. . According to this rule, the resistance moment in the given range of rotation speed was experimentally determined, and the torque was calculated based on the armature circuit current of the DC motor, and the flow curve was established in "resistance moment - angular velocity" coordinates.

The following mathematical expression aimed at evaluating the sensitivity of the device was obtained based on the separate consideration of the nominal (manufacturer's guarantee) values of the electrical and mechanical losses of the transmission:

$$\frac{\partial I}{\partial M} = \frac{\omega}{U_n\left(U_n^2 + 4(r_a + r_d)(M\omega + P_n(1-\eta)\right)^{\frac{1}{2}}}$$

The third chapter is devoted to the analysis of information indicators of viscometric measurements, their involvement in quality forecasting and management. Algorithms for approximation of anomalous flow functions in polymerization are developed here.

As the reaction mass conversion increases in periodic polymerization processes, the most important change in the environment is reflected in the shape of the fluidity curve. Figure 8 plots two flow functions (FF) for comparison.



a) b)

Fig . 8. Two fundamentally different forms of flow curves of polymer-based materials and characteristic parameters.

a) graph of dependence of the known tangential stress on sliding speed for pharmaseptic ointment, b) rotational-viscometric characteristic obtained from our experiment on epoxide resin.

In the figure, the graph (b) is taken from the results of our research, which is shown for comparison, and the graph (a) is taken from the literature.

In the study, the projection of the flow function into the three-dimensional space of initial moments is used as the main tool for parametrizing these functions. The initial moments of the 1st, 2nd and 3rd orders are written in the interval for the flow functions : $\omega \in [a,b]$

$$\mu^{(1)} = \int_a^b \omega \cdot \varphi(\omega) d\omega; \quad \mu^{(2)} = \int_a^b \omega^2 \varphi(\omega) d\omega; \quad \mu^{(3)} = \int_a^b \omega^3 \varphi(\omega) d\omega;$$

arbitrary non-stationary fluidity function $(\mu^{(1)} \times \mu^{(2)} \times \mu^{(3)})$ given in Fig. $\varphi_A(\omega,t)$ 9 is shown with the corresponding spatial projection ($A(t_0)$, points $A(t_1)$) and the corresponding trajectory.

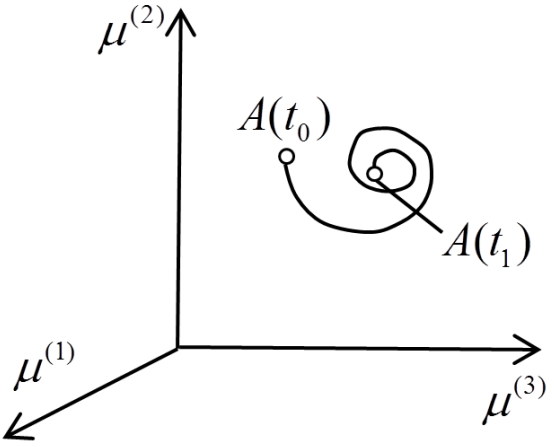


Fig. 9 . The projection of an instance of the stream function into the space of three ordered initial moments and the $t \in [t_0, t_1]$ trajectory drawn in space by its interval $A(t_1)$ point .

Measurement-calculation analysis of flow functions of simple polymer solutions

The simplest test among the algorithmic analysis tools we carry out in order to study the dependence of the process of polymer masses on the process of coming to the finished material form, in other words, on the trajectory drawn in the space of moments, is to obtain the rotational fluidity characteristics of mixtures of polymer samples with solvents in different proportions and how they are related to a number of physical quantities. is determined.

For this purpose, using epoxide resin as a polymer material, the relevant experimental results were analyzed based on it. Acetone was taken as the solvent, and the "acetone-resin" ratio was kept unchanged during the experiment in the predetermined variants.

The experiments were carried out based on a two-factor experimental design (temperature and concentration) and the results were analyzed in the form of a three-dimensional graphical representation (Figure 10 (a and b)).

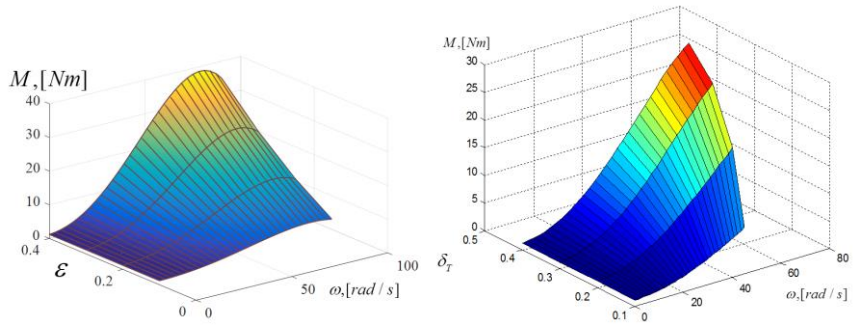
Taken as one of the coordinates in the graphs . $\omega,[rad/s]$ As other arguments, i.e. temperature and thickness coordinate axes corresponding to each graph are included. Those quantities are expressed as follows after centering and normalization:

$$\delta_T = \frac{T-\bar{T}}{\bar{T}}; \varepsilon = \frac{C-\bar{C}}{\bar{C}}, \quad (1)$$

where $T, C -$ the temperature at which the measurement is made and the concentration of the solution $\bar{T}, \bar{C} -$ are the averaged values of those quantities.

Measurements were made at the indicated values of temperature and solidity ($T = 25\ ^0C$; $\varepsilon = 0.1;$) in the respective variants. Changing the corresponding arguments is limited to 4 measurements:

($\varepsilon = \{0.1; 0.2; 0.3; 0.4;\}$; $\delta_T = \{0.1; 0.2; 0.3; 0.4;\}$).



a) b)

$\varepsilon = \{0.1; 0.2; 0.3; 0.4;\}$, $\delta_T = \{0.1; 0.2; 0.3; 0.4;\}$,

$T = 25\ ^0C$ in prices in $\varepsilon = 0.1;$ prices

Fig. 10 . Variation of moment of resistance as a function of rotation speed in viscometric measurement of epoxide resin dissolved in acetone.

The main conclusion we reached about the results of these measurements was that the flow curves themselves should carry enough information . As can be seen from the graphs, the measurements are highly sensitive to both temperature and concentration changes. This result leads to the conclusion that there is a big difference from the point of view of informativeness in constructing flow curves with simply measuring the viscosity under the conditions of a specified rotation speed. Thus, this fact significantly strengthens the a priori idea about the effectiveness of studying polymer masses in the space of fluidity functions in terms of quality prediction and management.

Partitioning of the set of flow curves into separate regions in the three-order initial moment space.

Although the fact that flow curves are highly sensitive to temperature and solvent effects by nature (in terms of shape) has created a valid basis for their inclusion as an argument in the prediction problem, accepting the examination of the degree of separation of clusters into separate regions as an additional analysis

method that can serve to further strengthen this basis can Again, referring to the use of epoxy resin as an experimental material, allowed by our technical capabilities, the picture of the injection of AFs into the space of moments was investigated.

To check the informativeness, the method of artificially "differentiating" the experimental materials was applied, and for this purpose, the same epoxy resin was mixed with different amounts of oak dust as the test material (with a weight ratio of 0.12 and 0.18) and a viscometric study was carried out.

Thus, the 1st and 2nd sample consisting of epoxy resin and oak powder is constructed as a viscometric measurement-research material under the condition of mixing the solvent in different proportions. The obtained experimental points are polynomially approximated, that is, a polynomial passing through 5 points with 5 variants in two groups of experiments is constructed:

$$M = \phi_{ik}(\omega, \varepsilon); 0 \le \omega \le \omega_{\max}; \varepsilon_{\min} \le \varepsilon \le \varepsilon_{\max}; i = 1, 2.$$

Graphic representations based on those polynomials are shown in fig. 11 is shown.

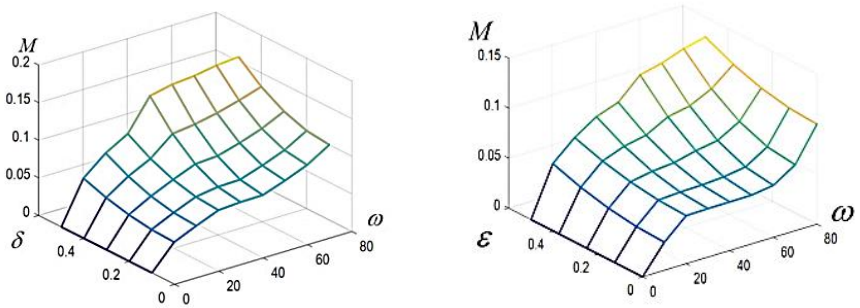


Fig. 11. Expansion and two-dimensional polynomial approximation of the fluidity curves established for two experimental samples according to the thicknesses taken in 5 variants.

Obtained approximations to the moment space $(\mu^{(1)}, \mu^{(2)}, \mu^{(3)})$ is moved. That is, the experiment is carried out by changing the solvent ratio and the corresponding number of experimental points in the moment space is obtained.

During the experimental studies, care was taken to ensure that the rotation speed intervals had the same width. To achieve this, the oak dust-resin ratio (weight ratio) should not exceed 0.2. At higher values of this ratio, a sharp increase in viscosity occurs, so the rotational resistance moment exceeds the intended upper limit.

Thus, as a result of experiments, fig. An image representing experimental points in three-dimensional moment space, reflected in Fig. 12, was obtained.

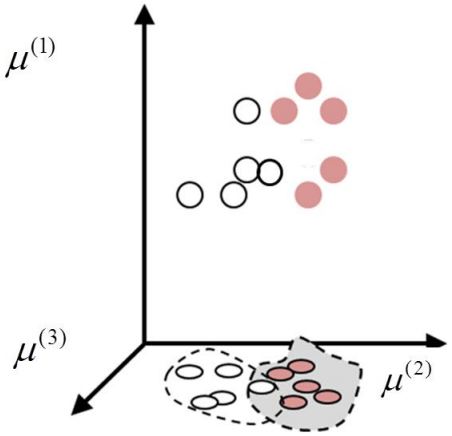


Fig. 12 . Representation of the set of flow curves in the corresponding set of three-dimensional starting moments.

Fig. An important conclusion obtained from the description given in Fig. 12 is that the set of flow curves in moment space is divided into separate subsets. This means that the flow curves carry enough information about the physical properties of the material, and if this information is processed in a proper way, those properties can be detected.

Table 1

Parameters of moment space analysis of experimental fluidity curves obtained for samples with different proportions of solvent (acetone) added

The temperature at which the experiments were carried out, $T = 25\ ^0C$;					
First example: Acetone-to-powder ratio =0.12					
Composite-acetone ratio $\varepsilon = m_s / m_r$	Values of moments			Total distance between points within a group	The distance between the centers of the groups
	$\mu^{(1)}$	$\mu^{(2)}$	$\mu^{(3)}$		
0.0	0.0615	0.0431	0.0322		
0.04	0.0518	0.0351	0.0227		
0.08	0.0478	0.0332	0.0225	0.102	
0.12	0.0461	0.0330	0.0216		
0.16	0.0442	0.0316	0.0145		
Second example: Acetone-to-powder ratio =0.18					
composite-acetone ratio $\varepsilon = m_s / m_r$	$\mu^{(1)}$	$\mu^{(2)}$	$\mu^{(3)}$		0.230
0.0	0.0517	0.0360	0.0239		
0.04	0.0467	0.0327	0.0268		
0.08	0.0447	0.0324	0.0188	0.097	
0.12	0.0440	0.0265	0.0108		
0.16	0.0416	0.0243	0.0103		

The following approximations are written as material to the computational experiment procedure:

$$M = \phi_k(\omega, \varepsilon),\ M = \phi_\ell(\omega, \delta),\ k = \overline{1, K};\ \ell = \overline{1, L} \qquad (2)$$

where the index *k represents variations due to the* amount of acetone, ℓ and the number of variations due to temperature.

Again, the stage of moment calculation according to these approximations is performed and the results are processed according to the following formulas:

$$k_{\varepsilon}^{2}=\frac{1}{K}\frac{1}{(\overline{\varepsilon}-\varepsilon_{k})^{2}}\sum_{k=1}^{K}\sum_{i=0}^{2}\left(\overline{\mu}_{k}^{(i)}-\mu_{k}^{(i)}\right)^{2};$$
$$k_{\delta}^{2}=\frac{1}{L}\frac{1}{(\overline{\delta}-\delta_{\ell})^{2}}\sum_{k=1}^{L}\sum_{i=0}^{2}\left(\overline{\mu}_{\ell}^{(i)}-\mu_{\ell}^{(i)}\right)^{2}; \quad (3)$$

where that k_{ε}^{2}, k_{δ}^{2} – correspondingly, it expresses the value of the sensitivity to the temperature variations on the scale of the moments vector, $\varepsilon_{k},\overline{\varepsilon}$ – the composite-acetone ratios taken in the experiments and the average value calculated for them, $\overline{\delta},\delta_{\ell}$ – the parameters expressing the values of the analogous indicators with respect to the temperature, according to the $\overline{\mu}_{\ell}^{(i)},\mu_{\ell}^{(i)};\overline{\mu}_{k}^{(i)},\mu_{k}^{(i)};i=0,2$ – relevant variations, 0,1,2- are the design moments.

It should be noted that the calculations once again confirm that the moment vector reacts not to the averaged ordinate of the flow curves, but to their shape, as they have sensitivity indicators and normalized according to their inclinations in k_{δ}^{2} formulas $(\overline{\delta}-\delta_{\ell})$($(\overline{\varepsilon}-\varepsilon_{k})$ 3) . k_{ε}^{2} This property once again proves that those formulas are effective enough for sensitivity estimation.

Setting the issue of indirect measurement .

It is assumed that there is a class of polymeric material suitable for rheological study (in sample number n), for each of which MMDFs were previously analyzed by laboratory method. It is accepted that the starting moments from the m-order were also calculated based on MMDFs. Those MMDFs are denoted by $\varphi_{i}(N);i=\overline{1,n}$, and the starting moments obtained based on $\mu^{(j)};\quad j=\overline{1,m}$ them.

Viscometric (rheological) experiments were carried out on those samples and *n* number of corresponding fluidity curves were obtained. Flow curves have *m* number of characteristic parameters and they are evaluated individually for each sample: $x_{ij};i=\overline{1,n};\ j=\overline{1,m}$. For example, according to the variant with three characteristic parameters, it will be as the numerical values of the variables $x_{i1}=\omega_{i1};x_{i2}=\omega_{i2};x_{i3}=\alpha_{i};\quad i=\overline{1,n}$.

The main hypotheses adopted in the matter:

1. The samples are materials of the same limited class, that is $x_i \in X \subset R^m$, their distinguishing features can only be the diversity of the technological regime allowed in production.

2. It is accepted that the rheological research was performed in the same apparatus, based on the same standard methodology.

3. Initially, a training phase is performed to ensure tuning of the deterministic algorithm. That is, the yield curves obtained from the experiment were obtained on the basis of those materials whose MMDF are a priori known functions, i.e. $x_k \to y_k; k = \overline{1,K}$, where x_k – the yield curves obtained from the experiment y_k – are moment vectors corresponding to the MMDF indicator of the material.

These hypotheses are intended as factors that ensure the accuracy of indirect measurement and evaluation.

Required: For an arbitrary sample, based on the known viscometric experimental results, determine the m – starting moments for the number of MMDFs with the $y_1 = \mu^{(1)}; y_2 = \mu^{(2)}; y_3 = \mu^{(3)}$ given $x \in X$ accuracy.

Let's first solve the problem based on the deterministic approach. For the sake of clarity, let's consider in the example of the three characteristic parameters described above, that is, in the solution of the three-dimensional problem. $x_{i1} = \omega_{i1}; x_{i2} = \omega_{i2}; x_{i3} = \alpha_i; \quad i = \overline{1,n}$ Let us assume that there is any operator A acting in a sufficiently limited domain where the parameters change, which maps X every element of its set Y to its set $A: X \to Y$. Consider this operator as a linear transformation operator:

$$Y = AX$$

where is $Y - (m \times 1)$ a column matrix of size 3- initial moments; X – in the same size, it is still a matrix of columns, and they reflect the characteristics of the electromechanical flow curve; and A – denotes the transformation matrix in size. $(m \times m)$

The solution of the problem will consist in determining that operator A, which can be considered as the simplest version of the deterministic approach.

For this purpose, let's choose three arbitrary options from experimental examples:

that is, let $\boldsymbol{y_i} = \{y_{1i}, y_{2i}, y_{3i}\}, i = 1,2,3$'s choose vectors such that their projections consist of the mentioned 1st, 2nd and 3rd order moments. As mentioned, these moments were obtained in advance based on the known MMDF. After conducting the rotation experiment of each sample separately, the corresponding $\boldsymbol{x_i} = \{x_{1i}, x_{2i}, x_{3i}\}; i = 1,2,3$ vectors are obtained.

If we pose the problem for a dimensional system X as a general case , then we have to write the following appropriate number $m-$of Y matrix blocks:

$$X = (\boldsymbol{x_1}\ \boldsymbol{x_2}\ \ldots \boldsymbol{x_m}); Y = (\boldsymbol{y_1}\ \boldsymbol{y_2}\ \ldots \boldsymbol{y_m})$$

It is known from the theory of linear operators that the linear operator projecting the system of initial vectors forming the basis to the X system of arbitrary Y vectors is defined as one-valued. This matrix can be obtained by constructing an inverse matrix.

Based on matrix algebra, it is possible to determine the converter matrix A as follows:

$$A = \begin{pmatrix} a_{11}\ a_{12} \ldots a_{1m} \\ a_{21}\ a_{22} \ldots a_{2m} \\ \vdots\quad\quad \ddots \\ a_{m1}\ a_{m2} \ldots a_{mm} \end{pmatrix} = \begin{pmatrix} y_{11}\ y_{12} \ldots y_{1m} \\ y_{21}\ y_{22} \ldots y_{2m} \\ \vdots\quad\quad \ddots \\ y_{m1}\ y_{m2} \ldots y_{mm} \end{pmatrix} \begin{pmatrix} x_{11}\ x_{12} \ldots x_{1m} \\ x_{21}\ x_{22} \ldots x_{2m} \\ \vdots\quad\quad \ddots \\ x_{m1}\ x_{m2} \ldots x_{mm} \end{pmatrix}^{-1}$$

Table 1 shows the numerical values of rotation experiments performed on different brands of epoxy resin. The number of experiments and the size of the vectors are taken equal to 3. Calculation of moments based on MMDFs for those oligomer molecules was calculated according to the empirical function taken in the form of a Gaussian function. In order to replace these

exemplary calculations with exact results, natural, wider laboratory-experimental opportunities are required, and the creation of such conditions in modern times does not face any fundamental difficulties.

Fig. 13 shows the block diagram of the procedure implemented for the identification of MMDFs in a real technological process according to the algorithm described above.

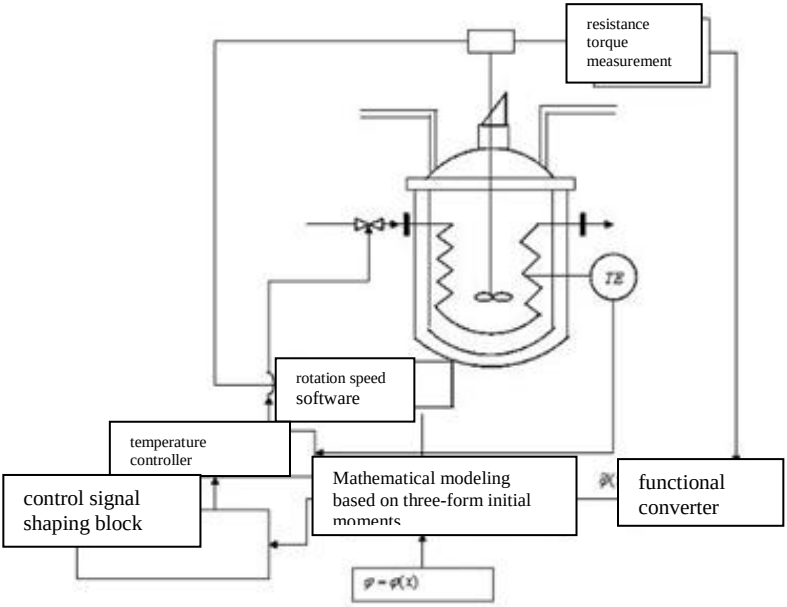


Figure 13 . Functional structure of the indirect determination system of the molecular mass distribution function

In the fourth chapter , optimal control algorithms are developed based on the state space concept for the periodic polymerization process. The concept of viscometric state space is included in the management of the periodic polymerization process.

Suppose that a $(\mu^{(1)}, \mu^{(2)}, \mu^{(3)})$ complex capable of reflecting the fundamental state of the polymerization process can be obtained by measurement at an arbitrary moment in time $0 < t < T$. Here $T -$, periodic is the period (period) intended for a complete course of the polymerization process. Assume that the projection of the molecular mass distribution functions into the space of three-component initial moments approximately satisfies the requirement of one-value within the given accuracy.

An important issue that attracts our attention in connection with the issue of the space of situations is the question of whether the dynamic system we are looking at has an " after-effect" or not . In order to interpret this effect and come to a conclusion , we had to include the concept of trajectory in the three-dimensional space that we introduced earlier. Fig. Some of those trajectories are reflected in 14 .

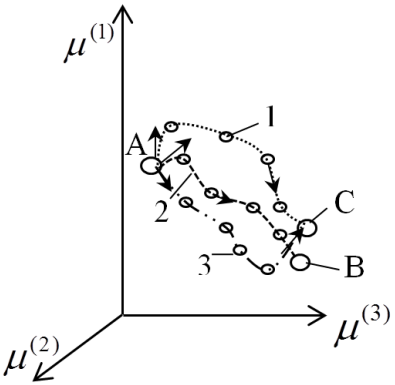


Fig. 14 . Description of three trajectories in moment space:
Curves 1 and 3 are bounded at point C, which is the same for both; The 2-curve is bounded by point B.

In the illustration in the picture, three trajectories from the same point decide on different points of space at different moments of time. Although the time coordinate is not included in the three-dimensional coordinate system, the time coordinate is also recorded

in the calculation-control system. The flow curves are treated as inflections of MMDFs, and the moments as parameters of the flow functions.

The exact answer to this question can be obtained, of course, based on the use of experimental methods. A logical answer refers to the proposition that molecular mass distribution functions are well established as qualitative characteristics of polymer masses. Thus, it is assumed that the space of viscometric states in polymerization has the following properties:

1. Each point in the space of moments drawn according to the flow functions has uniqueness as a specific qualitative characteristic of the polymer mass;

2. The quality is determined only by the coordinates of the point in that space, the path (trajectory) leading to those coordinates practically does not determine the quality.

These properties, we believe, lay the foundation for introducing the concept of optimal trajectory and solving the problem of quality management from it.

Reference trajectory, inclination and velocity vector in state space

Visualization of the flow functions in the space of moments and the change of this function depending on time is shown in fig. 15 creates the image given in

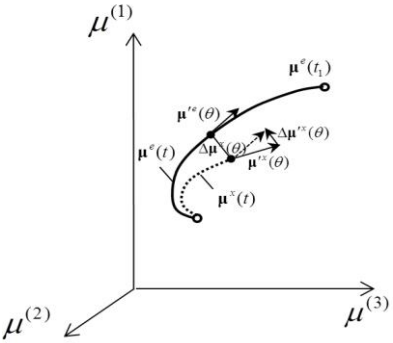


Fig. 15. Optimal and current trajectories in moment space, inclination θ and corresponding velocity vectors at arbitrary time

One of the main positions in the image is occupied by the standard trajectory, i.e. $\boldsymbol{\mu}^e(t)$ the curve marked with . The cross-section of the time when the process is finished t_1 is marked as in the graph. Marked as an arbitrary point in the interval over which time changes $\theta \in (0, t_1]$. In that graph, different from the optimal trajectory, another trajectory and its t_x moment $\boldsymbol{\mu}^e(t)$ the part indicating the inclination from the point is $\left\|\Delta\boldsymbol{\mu}^x(\theta)\right\|$ shown. The length of that piece is determined as follows:

$$\varepsilon = \left\|\Delta\boldsymbol{\mu}^x(\theta)\right\| = \sqrt{\sum_{i=1}^{3}\left(\mu^{e,(i)}(\theta) - \mu^{x,(i)}(\theta)\right)^2}$$

where $\mu^{e,(i)}(\theta), \mu^{x,(i)}(\theta)$ – denote the corresponding points on the time reference and current trajectories, respectively.

Let's state the issue of control in this way: it is necessary to determine the control effect corresponding to the inclination at an arbitrary θ moment $\Delta\boldsymbol{\mu}^x(\theta)$, so that this effect is proportional to the inclination and directed opposite to it.

Considering the fact that there is a control effect in the polymerization process in connection with the formulation of the problem in this way, u let us denote it by.

There is no doubt that the effect of the quantity we are looking at on the dynamics of the system will be an aperiodic transition process. u Taking this into account, there is a good reason to assume aperiodic response to the control effect in the arbitrary state of the system. To determine this reaction, Fig. Note that two more vectors are involved in the graph given in Fig. 15 and that they are velocity vectors. Note that these velocity vectors h are determined by the difference of the state vectors of the system in two close time intervals:

$$\boldsymbol{\mu}'^x(\theta) = \lim_{h\to 0}\frac{\boldsymbol{\mu}(\theta+h) - \boldsymbol{\mu}(\theta)}{h}.$$

Let's consider the modeling of the effect of the scalar on that $\mu^{e,(i)}(\theta), \mu^{x,(i)}(\theta)$ angle, based on the dependence of the angle between the vectors of the tilt. The u following formula was used to determine the angle between two vectors in three-dimensional vector space : ξ

$$\xi = \arccos \frac{\sum_{i=1}^{3} \mu'^{e,(i)}(\theta)\mu'^{x,(i)}(\theta)}{\left(\sum_{i=1}^{3}\left(\mu'^{e,(i)}(\theta)\right)^2\right)^{\frac{1}{2}} \cdot \left(\sum_{i=1}^{3}\left(\mu'^{x,(i)}(\theta)\right)^2\right)^{\frac{1}{2}}}$$

where $\boldsymbol{\mu}'^{e}(\theta)$, $\boldsymbol{\mu}'^{x}(\theta)$ - are the standard and current velocity vectors, respectively, in moment space.

After that, by relating the angle of ε inclination of the object to the standard and ξ the distance covered during the time L on the trajectory , we get the following expression for τ that ε quantity:

$$\varepsilon = L\sin\xi; L = \int_0^{\tau} \sum_{i=1}^{3}\left(\mu^{x,(i)}(\theta)\right)^2 d\theta \qquad (4)$$

After that, assuming that the switching process on the control channel is aperiodic, the corresponding switching process differential equation is written:

$$a_1 \frac{d\varepsilon}{dt} + a_2\varepsilon = 0; \quad \text{or } \frac{d\varepsilon}{dt} + a\varepsilon = 0$$

Consider the received expression as a switching process on the control channel, and the control effect as belonging to the class of rectangular pulses, that is:

$$u(t) = \begin{cases} 0, if\, t < 0 \\ 1, if\, t \geq 0 \\ 0, if\, t > 1 \end{cases}$$

Based on this, we can write the following equation for the transition process:

$$\frac{d\varepsilon}{dt} + a\varepsilon = u(t) \qquad (5)$$

The optimal trajectory problem that provides the desired quality

In recent years, the issue of obtaining materials with the desired quality has become very relevant in polymer technology, and it should be noted that it is preferable to solve this issue by considering molecular mass distribution functions. Since there is a close relationship between the flow function and the quantities reflecting its distribution in the space of moments, it can also be used successfully in this matter as well.

With reference to the general control concept, we can say that $t = t_1$ the development of the algorithm that provides approximation to the standard moment vector in the time section of the differential equation (5) is ahead . In that formula, the $\varepsilon(t)$ function , which is t_1 the tendency criterion, must approach zero at the instant of time and thereby satisfy the optimality criterion. At an arbitrary moment of time, we can write the following control equation proportional to the bias based on the principle of feedback:

$$\frac{d\varepsilon}{dt} + a\varepsilon = k(t)\varepsilon; \quad \text{or} \quad \frac{d\varepsilon}{dt} + (a - k(t))\varepsilon = 0; \tag{6}$$

Note that expression (6) actually represents the differential equation form of the traditional proportional feedback concept. Algorithmizing this approach does not create a fundamental difficulty. Moments should be measured at each time interval, the norm of the corresponding moment vector should be calculated, ε its inclination should be calculated according to the formula (4) and an effect that either accelerates or slows down the polymerization reaction should be applied according to its value and sign. Control based on the feedback mechanism is shown in Fig. The block diagram in Fig. 16 can be visualized with the given algorithm.

As it can be seen, the main issue of the control process is the determination of the displacement caused by the control effect to the vector of moments.

Let's note that the velocity vector in the space of three-dimensional moments is defined by the angle it forms with respect to the three coordinate axes in space. Let us assume that the control effect vector consists of reaction initiator, inhibitor and temperature. In such a case, re-applying to the following linear transformation operator will be justified. However, the three-dimensional regression dependence with higher efficiency can be obtained by the empirical modeling method.

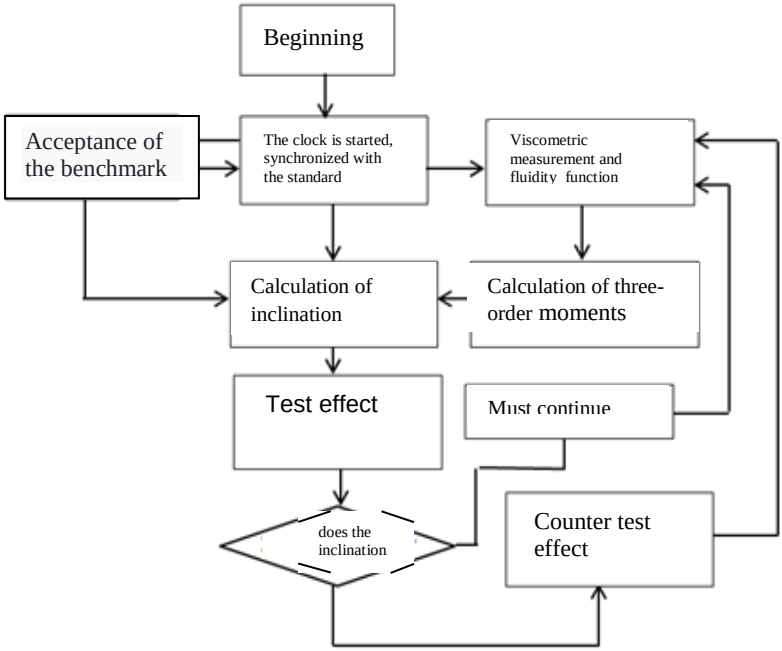


Fig. 16 . Block diagram of the approximation algorithm to the standard trajectory based on viscometric measurements during the polymerization process

The structure of the model referring to the regression dependence is described below.

$$\varphi_{1,2} = a_{11}u_1 + a_{12}u_2 + a_{13}u_3 + a_{14};$$
$$\varphi_{1,3} = a_{21}u_1 + a_{22}u_2 + a_{23}u_3 + a_{24}; \quad (7)$$
$$\varphi_{2,3} = a_{31}u_1 + a_{31}u_2 + a_{31}u_3 + a_{34};$$

where, $\varphi_{1,2};\varphi_{2,3};\varphi_{1,3}$ – respectively, the angle formed by the velocity vector with the coordinate axes, u_1, u_2, u_3 – and the relative amount of the initiator intended to increase the speed of the process (the amount of the reaction mass brought to a unit volume, the amount of inhibitors that inhibit the reaction rate, and the temperature of the reaction mixture.

Consideration of the transition process on the control channel

In the study, it was considered that the main reason why the impulse transition characteristic is rarely used as a control model in the management practice of technological processes with a large inertia property is the technical difficulty in empirical determination of this model, that is, large errors can be made in recording the weight functions experimentally. Therefore, it is necessary to bring the differential equation model (5) written for the transition process into the form of a weight function, and then express the response of the system in the form of Duhamel's integral based on it.

The Duhamel integral, as is known, can be written on the basis of the transfer function obtained for the linear system. When the input of each transfer function in the form of the Laplace operator is affected by a delta-impulse, the response received at the output of that linear system determines the weight function.

Obtaining the output response of the system by simply treating the delta pulse as a sufficiently narrow and high-amplitude pulse and describing it as a Laplace transform is currently easily solved by standard programs.

The external influence on the system, that is, the concentration of the initiator or inhibitor inside the polymerization reactor, and

their $\frac{d\delta}{dt} + a\delta = u(t)$ In connection with what we denote by in the model $u(t)$, we should note that in the considered case u_t' – we have the opportunity to use only the time derivative of the control effect, i.e. This argument, in turn, stipulates the use of the model written in the form of a dual integral of the control channel transition process. In this case, the reaction of the system to the control effect is expressed as follows:

$$y(t) = U(0)h(t) + \int_0^t U'(\xi)h(t-\xi)d\xi \qquad (8)$$

where is a function of the $U(0)$ – control effect (the initial concentration of the initiator in the reactor, $h(t)$ – the impulse transition characteristic on the control channel, $U'(t)$ – the time derivative of the control effect (mass velocity of the initiator flow supplied to the reactor), $y(t)$ – the change in the state of the system.

According to the differential equation (5) we have considered $W(p) = \frac{k}{\frac{1}{a}p + 1}$ The weight function corresponding to the transfer function in Fig. is as follows:

$$h(t) = k \cdot a \cdot e^{-at} \qquad (9)$$

where is $\frac{1}{a}$ – the time constant of the transition process on the influence channel, is k – the amplification factor of the channel.

Let's use expression (7) to determine the response to impulses acting on the system based on the expression of the channel with a model given in the form of a weight function : $h(t) = kae^{-at}$

$$\varepsilon(t) = ka\int_0^t e^{-(at-\xi)}u'(\xi)d\xi \qquad (10)$$

where that k, a – (10) parameters of the model (they are estimated by passing through the parametric identification stage), the deviation from the $\varepsilon(t)$ – standard trajectory $u'(t)$ – is a vector-function

reflecting the mass velocity of the initiator and/or inhibitor supplied to the reactor.

Based on the considered control concept, the version of the model problem designed for the example was solved in the MATLAB programming environment. In the problem formulated for the example, the cost of the initiator was taken as the control effect. A problem with a two-coordinate control vector will not differ from a scalar control effective problem only if the joint effect of both control factors has the property of additivity and bears the sign of orthogonality. Of course, it is not possible to achieve program control as in single-factor control in complex polymerization processes that do not meet the requirement of such a severe condition. In these cases, using the advantages of man-machine control systems can greatly simplify the issue and increase efficiency.

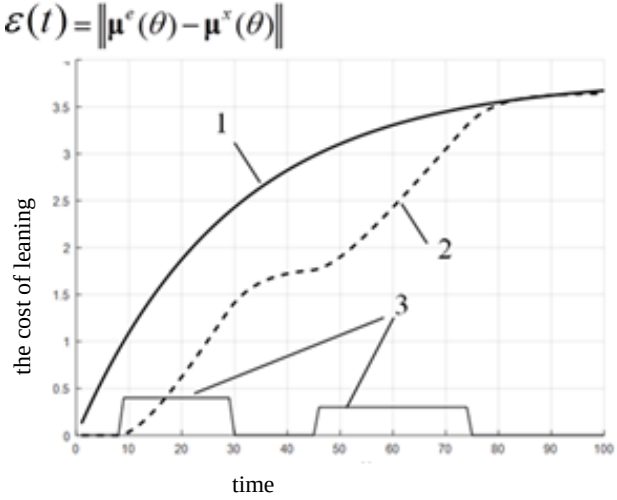


Fig. 17. Graphs depicting the approach to the benchmark trajectory with impulsive control effects

Fig. 17 shows the solution of the model problem for the case with a scalar control effect. The problem is formulated for a zero initial condition, and with the impulsive application of the control

effect, an example of a two-step transition process control in the system is obtained.

The initial supply of the initiator to the system was $t = (7-30)\,deq$ carried out in the form of a pulse covering a time interval. The amplitude of the impulse was kept constant during this time and was $A = 0.5 -$ taken equal to its value. As the effect of this pulse wears off, the polymer-forming reaction again decelerates appreciably, creating a curve in the graph. The impact of the next pulse in the time interval (45–75) increases the rate of polymerization again, and the process of approaching the standard trajectory appears.

THE MAIN RESULTS OF THE DISSERTATION WORK

1. The characteristics of polymerization processes were analyzed from the point of view of automatic control and management object, scientific information was summarized in the direction of solving the problem of quality control in the production of polymer masses. [1,4]
2. A comparative analysis of measurement and control methods applied in polymerization processes was conducted, and the perspectives of the issue of viscometric quality control were substantiated. [8,9]
3. The problem of indirect assessment of molecular mass distribution functions, which is an effective quality indicator of polymer masses, has been worked out mathematically and the prospects for the solution have been investigated. [5,6]
4. A method for approximation of molecular mass distribution functions of polymers by means of Gauss function parameters is proposed. .[3,7]
5. The issue of rotational rheological study of a non-Newtonian fluid medium with a polymer-based polydisperse structure was proposed and a method was developed for the evaluation of its informativeness indicators from the point of view of quality control. [10]

6. For the rheological study of the non-Newtonian polymerization reaction environment, the principle of rotational measurement was developed and its realization was achieved in the example of a laboratory measuring device. [15]
7. The method of parametrization of the empirical flow functions in the area of 3 sets of initial moments was developed and the method for the identification of quality indicators in the space of those parameters was developed. [9]
8. An algorithm for constructing a linear operator that creates a functional relationship between molecular mass distribution functions and vectors of quality indicators has been developed. [11]
9. Based on the measurement of the moment of resistance encountered by the polymerization reactor mixer transfer, the methodical bases for the operational rheological study of the reaction mixture were developed and the informativeness factor was evaluated. [12,13]
10. Based on viscometric measurements in the polymerization process, an early prediction and automatic control system algorithm of harmful gel-effect was developed. [17]
11. An optimal control algorithm for the periodic polymerization process was developed based on the state space concept. [16]
12. The problem of obtaining the desired quality and the optimal trajectory for its achievement in the polymerization process was formulated and a solution algorithm was developed. [14]

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17. Hashimova HM The issue of early prediction of the gel effect, which has a detrimental effect on product quality in polymerization processes. / Information systems and technologies achievements and perspectives. Proceedings of the II International Scientific Conference. 2020, p. 39-40.

Personal activity of the plaintiff in cases with co-authors:

[1] – Compilation of reporting programs and analysis of results;

[2,4,5] - Development of the algorithm of mapping the initial moment space to the desired parameters vector space.

[3,7] - Mathematical formulation of the problem of optimal control of the thermal regimes of the polymerization reactor and development of the solution algorithm.

[6] - Development of the algorithm for calculating the moment of viscous resistance in mixing transfer;

[9] - Sensitive element of the viscometer - calculating the moment of resistance encountered by the rotor and working out the principle of obtaining flow curves.

[10] - Development of the algorithm for solving the problem of measuring the resistance moment generated in the shaft of the mixer transmission of the polymerization reactor in the variable speed mode;

[13] - Mathematical formulation of the issue of optimal control of polymerization processes with quality criteria;

[15] - Solving the issues of evaluation of quality informativeness and quality prediction based on rheological research.

The defense will be held on “31” October 2022 at_16.00at the meeting of FD 2.25 Dissertation Council atttached of Sumgait State University.

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