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Analysis of the Physical and Chemical Processes of Formalin Production for Building a Decision Support System in Management

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Abstract. To create highly efficient formalin production control systems, it is necessary to comprehensively and thoroughly study technological processes using active experimental methods, identify physicochemical patterns of processes and develop optimization and control algorithms based on them. This article discusses physical and chemical models of formalin production processes for building a decision support system for managing these processes.
Keywords. Automation, control, algorithm, analysis, mathematical model, technological process, regression model.

INTRODUCTION

The description of the technological scheme is given in accordance with [3]. The method for the production of formalin from methanol consists in obtaining formaldehyde on a catalyst "silver on a carrier" at a temperature of 550 ℃ ÷ 700 ° C, followed by its absorption in water and rectification.

The formalin production process for one technological line consists of the following stages:

- obtaining a methanol-air mixture;
- synthesis of formaldehyde;
- absorption of formaldehyde to produce "raw formalin";
- rectification of "raw formalin".

The nodes common to all threads are:

- collection and processing of substandard and drained products,
- purification of gas emissions,
- combustion of absorption gases in a flare, heat supply, collection and pumping of condensate.

The auxiliary nodes are:

- preparation of the catalyst,
- storage and shipment of formalin,
- thermal waste disposal.

METHODS

Graphic representation of the stage of obtaining methanol-air mixtures and contacting stages are given on FYURA PF 000.000 ST.

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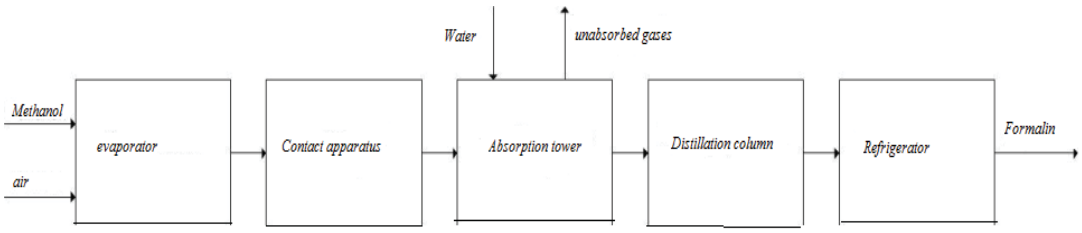

FIGURE 1. Block diagram of the formalin production process.

Formaldehyde is actually formed as a result of parallel reactions of simple and oxidative dehydrogenation of methanol

$$CH_3 0H \rightarrow CHgO + H_2 - 93.4\ KJ / mol\ ; \quad (1)$$

$$CH_3 0H + 1/2\ 0_2 \rightarrow CH_2 0 + HgO + 147.4\ KJ / mol. \quad (2)$$

Along with these reactions, a whole complex of side transformations occurs in the system. The total thermal effect of all reactions is positive and, under the conditions of the technological process, amounts to 90-110 KJ / mol . ([1]

The schematic diagram of the formalin unit is shown in Figure 2 .

Formaldehyde is obtained from methanol, dimethyl ether, natural and associated gases (gas of coke production and oil refining). Industrially mastered methods of obtaining formaldehyde: [2]

1) Catalytic oxidation of methanol on metal catalysts.

2) Catalytic oxidation of methanol on oxide catalysts.

3) Oxidation of natural gas and lower paraffins.

In the first case, silver is most often the catalyst (gold or platinum can be used); in order to save methanol, silver is uniformly applied to an inert carrier, for example, pumice. The essence of the method consists in the vapor-phase oxidation of methanol hydrogenation with atmospheric oxygen in an adiabatic reactor, followed by the absorption of the reaction products by water. In the process, a methanol-air mixture is supplied with a composition above the upper explosive limit (36.4% - volume fractions) and with oxygen deficiencies according to the chemical equation for the oxidation of methanol to formaldehyde, since catalyst - oxidation-dehydrogenation:

$$CH_3OH + ½O_2 \rightarrow CH_2O + H_2O + Q\ (147.4\ kJ / mol); \quad (3)$$

$$CH_3OH \rightarrow CH_2O + H_2 - Q\ (93.4\ kJ / mol); \quad (4)$$

$$H_2 + ½O_2 \rightarrow H_2O + Q\ (241.8\ kJ / mol) \quad (5)$$

A mixture of methanol L_1 and water L_2 enters the alcohol evaporator $_1$, where it evaporates at a temperature of 338-360 K. After the alcohol evaporator, the alcohol-water mixture G_1 is mixed with air G_2 and, heated in heat exchanger 2 to a temperature of 383-413 K, in the form of an alcohol-water-air mixture G_3 enters the contact device 3, where on the catalyst 4 at a temperature of 923-1023 K, formaldehyde is synthesized. The contact gases $G_{4,\ having}$ cooled in the under-contact refrigerator 5 to a temperature of 393-413 K, enter the absorption stage. Gases G_4 G_5 G_6 successively pass through absorbers 6, 7, 8 and are removed to the G 7 flare . The absorbers are irrigated with a weak formalin L_3 , a poor formalin L 4 condensate L 5 and a circulating liquid L_6 , L_7 , L_8 . The recirculated liquid is cooled in remote heat exchangers 9, I0, 11 with the refrigerant L_9 , L_{10} , L_{11} . The finished 37% formalin L_{12} is transferred to the stockpile. The process is carried out under a slight overpressure of 1.65 * 10^5 Pa by continuous technology. The main operating mode of the unit is static.

The unsteadiness of the characteristics of the formaldehyde synthesis process due to a decrease in the activity of the catalyst, which leads to a decrease in the mass fraction of formaldehyde in the product. The decrease in the activity of the catalyst is compensated by an increase in the temperature of the contact apparatus

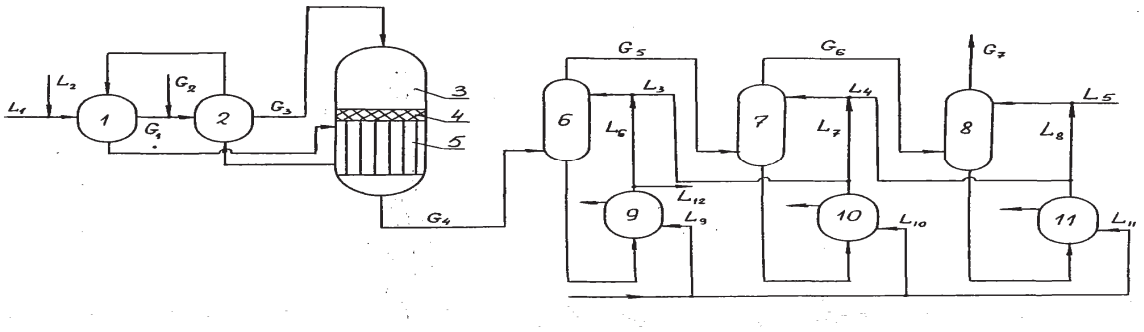

FIGURE 2. Schematic diagram of the formalin unit: 1 - alcohol evaporator; 2-heat exchanger; 3-pin device; 4-catalyst; 5-contact refrigerator; 6, 7, 8-absorbers; 9, 10, 11-sided heat exchangers.

up to 983 K; if it is impossible to obtain a conditioned product at a given temperature, the catalyst is subjected to regeneration.

Thus, the technological processes for the production of formalin are characterized by continuity, toxicity of raw materials, products and waste gases, fire and explosion hazard, a large number of technological parameters, drift of parameters of the formaldehyde synthesis stage, and a long residence time of absorbed components in absorption devices.

Mathematical models of chemical technological processes can be divided into two groups: theoretical physicochemical models and empirical statistical models.

It is proposed to use typical physicochemical models in the mathematical description of the contacting process, which makes it possible to obtain a simple and sufficiently accurate model for further use in the development of optimization and control algorithms.

Empirical models of technological processes can be used directly in adaptive algorithms for optimization and control of formalin production.

Let us consider the characteristic features of mathematical models of the first group regarding the production of formalin. In works ([4], a mathematical model of the formaldehyde synthesis process is described, which is a system of equations for material and heat balances on the catalyst grain:

$$-\omega\frac{\partial X}{\partial 1}+b(Y-X)=e\frac{\partial T}{\partial t}; \tag{6}$$

$$-\omega\frac{\partial T}{\partial 1}+\frac{\delta}{C_p}S_{уд}\left(©-T=e\frac{\partial T}{\partial t}\right); \tag{7}$$

$$b(X-Y)-W=e_K(1-e)\frac{\partial Y}{\partial t}; \tag{8}$$

$$\frac{\lambda_K}{C_p}\frac{\partial^2©}{\partial 1^2}-\frac{a}{C_p}S_{уд}(©-T)+\frac{1}{C_p}\sum_{j=1}^{m}(-\Delta H_j)W_j==\frac{C_K}{C_p}(1-e)\frac{\partial ©}{\partial t} \tag{9}$$

where ω is the linear velocity of the gas flow; X is the vector of component concentrations in the flow; Y is the vector of near-surface concentrations; W is the vector of the conversion rates of the components; T is the temperature in the flow; © - catalyst grain temperature; ΔH_j, j = 1, m is the thermal effect of the j-th reaction; C_K is the heat capacity of the catalyst; C_P is the heat capacity of the gas; a is the coefficient of heat transfer from the grain surface to the gas flow; - coefficient of thermal conductivity of the catalyst layer; S_{beats} - specific surface of the granular layer; b - coefficient of mass transfer; e_K is the porosity of the catalyst grain; e is the porosity of the catalyst layer.

The system of equations (Algorithm 3) was used to optimize the process of incomplete oxidation of methanol to formaldehyde in a reactor with a fixed bed of a pumice – silver catalyst [5] . In a number of works [8, 9, 10], he developed a model of a combined reactor for the oxidation of methanol to formaldehyde, taking into account the presence of an uneven velocity profile, temperature and concentration of gas flow components at the entrance to the contact layer, as well as the presence of heterogeneities in the permeability and catalyst activity in the layer:

$$\frac{\partial P}{\partial Z}=ueB;\ \frac{\partial P}{\partial X}=veB;\ \frac{\partial}{\partial Z}(upe)+\frac{\partial}{\partial X}(vpe)=0; \tag{10}$$

$$\lambda_Z\frac{\partial^2 T}{\partial Z^2}+\lambda_X\frac{\partial^2 T}{\partial X^2}-C_p\frac{\partial}{\partial Z}(upeT)-C_p\frac{\partial}{\partial X}(vpeT)+(1-e)\sum_{j-1}^{m}(-\Delta H_j)W_j=0; \tag{11}$$

$$D_Z\frac{\partial^2 C_i}{\partial Z^2}+D_X\frac{\partial^2 C_i}{\partial X^2}-\frac{\partial}{\partial Z}(ueC_i)-\frac{\partial}{\partial X}(veC_i)+(1-e)\sum_{j=1}^{m}(V_{ij}W_j)=0;\quad (I.4) \tag{12}$$

$$p=\frac{p_0}{[1+0{,}0036(T-T_0)]};\quad M=M_0\,\frac{T_0-111}{T+111}\left(\frac{T}{T_0}\right)^{3/2}; \tag{13}$$

$$B=\frac{150M(1-e)^2}{d_3^2e^3}+\frac{1{,}75(1-e)}{d_3}\sqrt{(ue)^2+(ve)^2}; \tag{14}$$

where i is the index of the substance; j is the reaction index; P - pressure; z, X - longitudinal and transverse coordinates of the layer; u, v - longitudinal and transverse velocities; P_0, P - the density of the reaction mixture, the initial and in the catalyst bed; D, - effective coefficients of diffusion and thermal conductivity, respectively; T_0, T is the initial temperature of the reaction mixture and in the catalyst bed; C_i - the concentration of the components of the reaction mixture in the catalyst bed; V_{ij} - stoichiometric coefficient of the i-th substance in the j- th reaction; M_0, m is the initial viscosity of the reaction mixture and in the catalyst bed; d_3 - catalyst grain diameter.

The system of equations (Algorithm 6) was used to calculate the parametric sensitivity of tubular and combined reactors, as well as to assess the influence of a wide class of spatial inhomogeneities on the characteristics of catalytic processes in apparatus of this type .

In [11, 12 :], the authors proposed a universal mathematical model for predicting the behavior of experimental packed reactors for the partial oxidation of methanol to formaldehyde, which is then used as a reference for comparing the effectiveness of various forms of models in practical reactor modeling:

$$e_p\frac{\partial C_S}{\partial t}=D_C\frac{1}{x^2}\frac{\partial}{\partial x}\left(X^2\frac{\partial C_S}{\partial x}\right)-R'(C_S,T_S); \tag{15}$$

$$(1-e_b)(pC_p)_S\frac{\partial T_S}{\partial t}=(1-e_b)\lambda\frac{1}{x^2}\frac{\partial}{\partial x}\left(X^2\frac{\partial T_S}{\partial x}\right)+k_{SZ}\frac{\partial^2 T_S}{\partial z^2}+k_{sr}\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T_S}{\partial r}\right)+$$
$$(1-e_b)(-\Delta H)R'(C_S,T_S); \tag{16}$$

$$e_b\frac{\partial C_f}{\partial t}=D_Z\frac{\partial^2 C_f}{\partial z^2}\;D_r\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial C_f}{\partial r}\right)-\frac{\partial(C_f v)}{\partial z}+S_v k_m(C_u-C_f);$$

$$e_b(pC_p)_f\frac{\partial T_f}{\partial t}=k_{fz}\frac{\partial^2 T_f}{\partial z^2}+ \tag{17}$$
$$k_{fr}\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial T_f}{\partial r}\right)-\frac{\partial(vpC_p)_f}{\partial z}+S_v h_{fs}(T_u-T_f);$$

where e_p is the porosity of the catalyst grain; C_S - concentration in the solid phase; D is the effective diffusion coefficient; t is time; x is the coordinate of the catalyst grain; R is the reaction rate; T_S is the catalyst temperature; e_b is the porosity of the catalyst layer; - coefficient of thermal conductivity of the catalyst grain; - coefficient of axial thermal conductivity of the catalyst layer; z is the axial coordinate of the reactor; P_S is the density of the catalyst; C_{ps} is the heat capacity of the catalyst; r is the radius of the reactor; k_{sr} - coefficient of radial thermal conductivity of the catalyst layer; ( ) is the heat of reaction; With $_f$ - concentration in the gas phase; D_z - effective axial diffusion coefficient; v is the volumetric velocity; k_m is the coefficient of mass transfer; With $_u$ - surface concentration; p_f is the density of the gas phase; C_{pf} - heat capacity of the gas phase; k_{fz} - coefficient of axial thermal conductivity of the gas phase; k_{fr} is the coefficient of radial thermal conductivity of the gas phase; h_{fs} - coefficient of interfacial heat transfer; T_u is the catalyst surface temperature; T_f is the temperature of the gas flow.

There are also works on modeling the processes of alcohol evaporation [13] and absorption [3, 15] In [15], a method for finding transfer functions of the third and fourth order was proposed for modeling a formaldehyde synthesis reactor.

The analysis of the theoretical mathematical models described above showed that the research results from the point of view of their control [3,15] were used to develop control algorithms:

1. The models quite fully reflect the basic physicochemical regularities of formalin production processes and in the absence of disturbances (decrease in the activity of the regenerated catalyst, changes in the quality indicators of methanol supplied to the alcohol evaporator, etc.) are adequate to the described processes.

2. Due to the fact that perturbations - a decrease in catalyst activity, changes in methanol quality indicators, etc. constantly affect the process of formalin production, the model must either take them into account explicitly,

which significantly complicates the mathematical description of the chemical-technological processes of formalin production, or use the algorithm for adapting the model parameters (preexponential factors, activation energies, and stoichiometric coefficients) according to the experimental data. These computational operations are very laborious both because of the high dimension of the problem and because of the nonlinear dependence of the parameters of the model; therefore, theoretical mathematical models, in the general case, are inappropriate to use in control algorithms; they should be used for designing devices, optimizing technological schemes, developing simplified management models.

A characteristic feature of empirical mathematical models of formalin production technological processes is the following. In connection with the above-mentioned complexity of chemical-technological processes for the purposes of control and optimization of production, they are often limited to their simplified description in the form of finite equations. At the same time, a mathematical description of the static modes of production is considered. In general, the target optimality criterion and control the process of obtaining the formalin Y - yield of formaldehyde is dependent on a number of process variables X_1, X_2, ... X_N. There is a general functional relationship between them:

$$Y = f(X1, X2, \dots Xn) \tag{18}$$

admitting a Taylor series expansion:

$$Y = b_0 + \Sigma_{i=1}^{n} b_i X_i X_j + \Sigma_{i=1}^{n} b_i X_i^2 + \dots, \tag{19}$$

where Y is the objective function; Xi, Xj - process parameters; b_O, b_i, b_{ij}, b_{ii} - regression coefficients.

When optimizing and controlling technological processes of formalin production, both empirical equations based on an expression (Algorithm 14) and regression dependences based on the expansion of the objective function in a Taylor series (Algorithm 15) are used. The first group includes works by J. Walker [18] and G. T. Shcherban [19] on determining the yield of formaldehyde depending on the composition of absorption gases: Shcherban's formula:

$$Y = \frac{1{,}05C_{H_2} + 0{,}563C_{N_2} - 3{,}07C_{CO_2} - 2{,}16C_{CO} - 0{,}8C_{CH_4} - 2{,}1C_{O_2}}{1{,}12C_{H_2} + 0{,}6C_{N_2} + 0{,}4C_{CH_4} - 2{,}15C_{CO_2} - 1{,}17C_{CO} - 2{,}25C_{O_2}} * 100 \tag{20}$$

(sixteen)

Walker's formula:

$$Y = 0{,}9375\ (100 - 100(\frac{C_{CO_2} - C_{CO} - C_{CH_4}}{2C_{CH_4} + C_{H_2} + 0{,}528C_{N_2} - 2C_{CO_2} - C_{CO} - 2C_{O_2}})) \tag{21}$$

where C_i, 1 = 1.6 - Content i- r o component in absorption gases. Formulas (1.8), (1.9) were used to determine the optimal operating modes of reactors and in the formalin production control system [20]

Among the works using their regression models of the form (15), one should note the work [2 1], in which a model is proposed that relates the formaldehyde yield to the oxygen / methanol ratio at the inlet to the contact apparatus:

$$Y = K_O + K_1 X \tag{22}$$

where Y is the formaldehyde yield; X is the oxygen / methanol ratio; K_O, K_1 - regression coefficients.

To adapt the model (alg 18) to a decrease in catalyst activity and other disturbances of the technological process, the Kachmazh algorithm was used [7,24]:

$$K_O[n] = K_O[n-1] + \frac{Y[n] - (K_O[n-1] + K_1[n-1]X[n])}{(Y[n] - \bar{Y}[n])^2 + 1 + (X[n])^2}; \tag{23}$$

$$K_1[n] = K_1[n-1] + \frac{Y[n] - (K_O[n-1] + K_1[n-1]X[n])}{(Y[n] - \bar{Y}[n])^2 + 1 + (X[n])^2} * X[n]; \tag{24}$$

$$\bar{Y}[n] = \frac{n-1}{n}\bar{Y}[n-1] + \frac{1}{n}Y[n], \quad n = 1,2,3, \dots, \tag{25}$$

where is the number of the control cycle;

Y is the current value of the formaldehyde yield;

is the average value of the formaldehyde yield.

Algorithm (18) together with the algorithm for adaptation of coefficients, algorithm (1 9) was applied for optimal control of formalin production through the channel "oxygen / methanol-formaldehyde yield" [2 1].

Analysis of empirical mathematical models of chemical-technological processes from the point of view of the possibility of their application in automated control systems for the production of formalin shows:

1. The models do not reflect the physicochemical laws of the formalin production process as fully as the theoretical physicochemical models. Empirical models are adequate to the described process only in a narrow range of parameter variation.

2. The adaptation of the parameters of these models is not difficult, for which, for example, regression methods of identification are used. In addition, the observed indicators of the quality of raw materials and catalyst can be included in the model in an explicit form without significantly complicating it.

Thus, empirical models of chemical technological processes can be used to optimize and control the direct production of formalin in flexible algorithms, while theoretical mathematical models use instrumental and technological process design, and simplified control models in the development of such algorithms can be used for improvement. got it before. We propose algorithms and mathematical models for making decisions under conditions of uncertainty using empirical models.

In terms of physical and chemical parameters, technical formalin must comply with the requirements and standards specified in Table 1.

TABLE 1. Norms of technical formalin requirements for physical and chemical indicators

The name of indicators	Norm for the brand FM GOST 1625-89	
	Top grade OKP 241731 0120	**First grade OKP 241731 0130**
1. Appearance	Colorless transparent liquid. During storage, the formation of turbidity or a white precipitate, soluble at a temperature not exceeding 40 ° C, is allowed.	
2. Mass fraction formaldehyde,%	37.2 + 0.3	37.0 + 0.5
3. Mass fraction methanol,%	4 - 8	4 - 8
4. Mass fraction of acid lot in terms of formic acid %, no more	0.02	0.04
5. Mass fraction of iron,%, no more	0.0001	0.0005
6. Mass fraction of the residue after calcination,%, no more	0.008	0.008

CONCLUSION

The physical properties of technical formalin depend on the content of formaldehyde and methanol in it and vary within:

- Density, kg / m ^{3} 1077 - 1116.
- Boiling point, ^{about} C 98.9.
- Specific heat, J / (kg K) 3352.
- Viscosity, cP 2.45 - 2.58.

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