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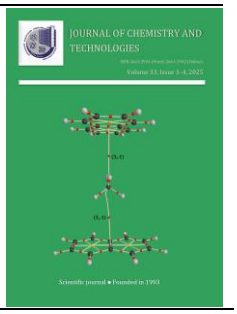

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ENHANCING THE PROCESSING EFFICIENCY OF LIGHT PYROLYSIS RESIN: A MATHEMATICAL MODEL APPROACH

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Abstract

The research work is devoted to enhancing the processing efficiency of light pyrolysis rezin for ethylene production and developing a mathematical model of this process. The process is aimed at increasing the yield of benzene by dealkylation of benzene-alkyl derivatives in the system using a toluene-based aluminum complex catalyst. Calculations were performed using the OptimMe and OriginLab software packages. By studying the effect of various factors (the amount of benzene at the beginning, temperature, the amount of catalyst, the duration of the process, etc.), the optimal temperature regime of the process, the composition and amount of the catalyst, and the duration of the process corresponding to the maximum yield of benzene were selected. Using these results, a multifactorial mathematical model of the process was developed, the adequacy of the model was determined by the Fisher's criterion, and the regression coefficients were evaluated by the Student's criterion. The obtained regression enables us to estimate the yield of benzene based on any concentration of the catalyst and the temperature conditions of the process (ranging from 0 to 80 °C). and thus can be used to minimize the total costs of implementing the process. Calculations indicate that the maximum yield of benzene occurs at a temperature of 80 °C, with a catalyst concentration of 2.5 %, and a process duration of 240 minutes.

Keywords: Student and Fisher criteria; mathematical modeling; light resin; benzene; metal-organic complex catalyst; regression model; dispersion; correlation.

ПІДВИЩЕННЯ ЕФЕКТИВНОСТІ ПЕРЕРОБКИ ЛЕГКОЇ ПІРОЛІЗНОЇ СМОЛИ: ПІДХІД НА ОСНОВІ МАТЕМАТИЧНОЇ МОДЕЛІ

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Анотація

Науково-дослідна робота присвячена підвищенню ефективності переробки легкого піролізного резина для виробництва етилену та розробці математичної моделі цього процесу. Процес спрямований на збільшення виходу бензолу шляхом деалкілування бензол-алкільних похідних в системі з використанням алюмінієвого комплексного каталізатора на основі толуолу. Розрахунки виконано з використанням програмних пакетів OptimMe та OriginLab. Вивчаючи вплив різних факторів (кількість бензолу на початку, температура, кількість каталізатора, тривалість процесу тощо), підібрано оптимальний температурний режим процесу, склад і кількість каталізатора та тривалість процесу, що відповідають максимальному виходу бензолу. На основі отриманих результатів розроблено багатофакторну математичну модель процесу, адекватність моделі визначено за критерієм Фішера, а коефіцієнти регресії оцінено за критерієм Стьюдента. Отримана регресія дозволяє оцінити вихід бензолу за будь-якої концентрації каталізатора та температурних умов проведення процесу (в діапазоні від 0 до 80 °C) і, таким чином, може бути використана для мінімізації загальних витрат на реалізацію процесу. Розрахунки показують, що максимальний вихід бензолу відбувається при температурі 80 °C, концентрації каталізатора 2/5 % і тривалості процесу 240 хв.

Ключові слова: Критерії Стьюдента і Фішера; математичне моделювання; легка смола; бензол; металоорганічний комплексний каталізатор; регресійна модель; дисперсія; кореляція.

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Introduction

The organic chemical industry has undergone a long and complex development process, resulting in radical changes in terms of its raw material base. It all began with the processing of plant and animal raw material, and then eventually transformed into coking, taking the form of the modern petrochemical industry, which has long since ceased to be satisfied with just petroleum refinery waste [1].

Oil-deep processing methods are classified into physical and chemical processes. Chemical methods are further divided into thermal and catalytic processing. Thermal processes include thermodestruction (thermal cracking, visbreaking, coking, pyrolysis, technical carbon production) and thermooxidation (bitumen production, gasification of coke and coal). Pyrolysis is crucial for the petrochemical industry, serving as the primary method for producing low molecular weight olefins like ethylene and propylene [2–9].

During the pyrolysis process, along with the main products, liquid by-products are formed [10], which, depending on the type of raw material and the mode of the pyrolysis process, constitute 25–35 % of the total amount of products. Liquid products are divided into two types: light and heavy resins. Light resin is rich in highly valuable components, and there are many methods for processing it.Resins are mainly used in the production of valuable products (aromatic hydrocarbons) and petroleum polymer resins, as their composition is rich in aromatic and unsaturated hydrocarbons [11–18].

There are many ways to process light resin, and the processing methods are based on its rectification from a mixture of benzene or its homologs, or its processing into petroleum polymer resin, which is a useful raw material for the paint and varnish industry.Most processes are based on multi-stage and costly technologies and the use of expensive catalysts (Pt, Pd) [19–24].

The main goal of the research work is to develop an efficient process for processing light resin, optimize it and provide a suitable mathematical model of the process. An efficient processing method involves a milder processing regime - conducting the process at lower temperatures and pressures and using cheaper catalysts.

Experimental part

In preliminary studies, the composition of the light resin was studied in detail. Light resin samples were analyzed using an Agilent GC 7820A system, and detailed compositional information based on these analyses is given in Table 1.

Table 1

Composition of several samples of light resin (mass fraction of components in light resin,%)

Hydrocarbons	light resin-1	light resin-2	light resin-3	light resin-4
Paraffin, olefin, naphthene				
C_5	1.36	3.48	3.44	0.87
C_6	8.10	14.6	17.83	10.39
C_7	0.18	0.15	0.15	0.19
Aromatic				
Benzene	39.48	41.33	37.12	32.84
Toluene	17.98	13.96	15.08	18.03
Ethylbenzene	0.93	0.97	0.94	1.17
xylene	3.85	1.68	2.84	3.75
styrene	7.94	6.56	6.51	8.65
1,3,5 trimethylbenzene	1.76	0.91	1.12	2.68
1,2,4 trimethylbenzene	6.75	7.47	5.91	9.17
1,2,3 trimethylbenzene	1.80	0.86	1.90	3.05
Identifiable	90.13	91.97	92.83	90.79
Unidentified hydrocarbons	9.86	8.02	7.16	9.20
total	99.99	99.99	99.99	99.99

For the research, light resin sample No. 3 was used. An aluminum complex catalyst based on toluene was initially prepared. At the initial stage, the amount of catalyst and the duration of the experiment were kept constant, and the processing process was carried out at different temperatures. The optimal temperature for the processing process was 80 °C. Then, keeping the other two parameters constant, experiments were carried out with different amounts of catalyst and at various time intervals, and the optimal catalyst time and optimal contact time (2.5 % and 240 min) were studied. These parameters were determined based on the maximum increase in

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benzene during treatment. The experiments were carried out in a reactor equipped with a heater, stirrer, thermometer, and anti-cooler; the structural diagram of the experimental setup is shown in Fig. 1.

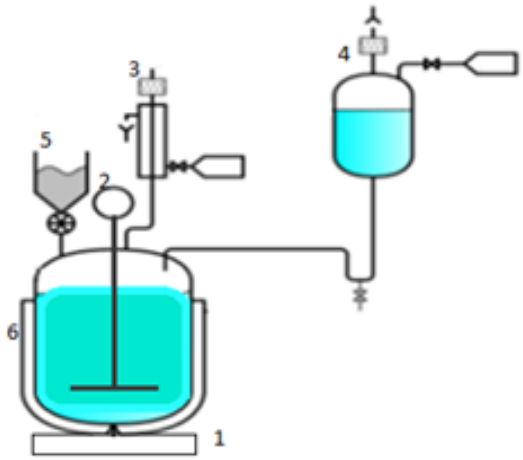

Fig. 1. Laboratory setup diagram 1. Heater, 2. Mixer, 3. Countercooler, 4. Receiver (fraction), 5. Separating funnel (catalyst) 6. Reactor (light resin)

The catalytically treated light resin was sent to a 3-stage rectification process (the last stage is azeotropic expulsion), and as a result, benzene with a purity of 99.2 % was obtained. Since the catalyst used in the refining process is a multifunctional catalyst, it causes reactions to occur in several directions in the system, including dealkylation, polymerization, oligomerization, and alkylation. The formation of benzene occurs due to the dealkylation of its alkyl derivatives:

$C_6H_4R_3 \rightarrow C_6H_4R_2 \rightarrow C_6H_4R_1 \rightarrow C_6H_6$

On the other hand, the intensification of polymerization reactions also clears the light resin of unsaturated hydrocarbons, and the bottom residue is enriched with resin. The remainder obtained from the rectification process is used to purchase petroleum-polymer resins.

To determine the optimal regime, experiments were conducted at different temperatures, different catalyst amounts, and different contact times, and the results are given in Table 2.

Table 2

The results of the preliminary experiments (technological regimes and benzene yield, %)

Experiment, №	Yield, %	Temperature, °C	Amount of catalyst, %	Duration of experiment, min.
1	39	10	4	60
2	39.15	15	4	60
3	39	40	4	60
4	39	60	4	60
5	40	80	4	60
6	38.5	100	4	60
7	43.085	80	2	60
8	41.13	80	3	60
9	45	80	2.5	120
10	47	80	2.5	180
11	47.5	80	2.5	240
12	38	80	5	60
13	39	80	4	15
14	42	80	4	120
15	42.5	80	4	180
16	43	80	4	240
17	45	80	1	60
18	42	80	2.5	60

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Result and discussion

Correlation and regression analysis methods are widely used to search for dependencies between random variables. y and x_1, x_2, ... x_k experimentally study the dependencies between random variables, and a certain number of (sufficiently representative) independent experiments are carried out, which are calculated using regression analysis methods.

To create a model, we use the multiple correlation method of the regression equation of the form (1)

$$\hat{y} = b_0 + b_1 x_1 + b_2 x_2 + \ldots + b_k x_k \quad (1)$$

This equation describes the regression surface for k = 2 and the hypersurface for k > 2. This surface is called the response surface. For this study, three factors will be included in the regression equation – temperature, amount of catalyst, and duration of the experiment, and the relationship will be as follows (2):

$$\hat{y} = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 \quad (2)$$

Calculations were performed using the software packages Optim ME, Origin Lab, and Excel [25; 26].

We move from the natural data scale to a new one, normalizing random variables according to the following equations (3) and (4):

$$y_i^0 = \frac{y_i - \bar{y}}{S_y}; \quad (3)$$

$$x_{ji}^0 = \frac{x_{ji} - \bar{x}_j}{S_{x_j}}, \quad (4)$$

I =1, 2, 3...., n; j = 1, ..., k

y_i^0, x_{ji}^0 – here these are the normalized values of the relevant factors;

$\bar{y}, \bar{x}$ – average values of factors; S_y, S_{x_j} – standard deviations of coefficients are is calculated using formulas (5), (6):

$$S_y = \sqrt{\frac{\sum_{i=1}^{n} (y_i - \bar{y})^2}{n-1}}; \quad (5)$$

$$S_{xj} = \sqrt{\frac{\sum_{i=1}^{n} (x_{ji} - \bar{x}_j)^2}{n-1}}. \quad (6)$$

Calculated values of standard deviations of coefficients are shown in Table 3.

Table 3

Standard deviations of coefficients

Experiment. №	$(y_i - \bar{y})^2$	$(x_{ji} - \bar{x}_1)^2$	$(x_{2i} - \bar{x}_2)^2$	$(x_{3i} - \bar{x}_3)^2$
1	7.071167361	3633.410494	0.37345679	1406.25
2	6.295917361	3055.632716	0.37345679	1406.25
3	7.071167361	916.7438272	0.37345679	1406.25
4	7.071167361	105.632716	0.37345679	1406.25
5	2.752834028	94.52160494	0.37345679	1406.25
6	9.980334028	883.4104938	0.37345679	1406.25
7	3.207084028	94.52160494	1.929012346	1406.25
8	1.957667361	94.52160494	0.151234568	1406.25
9	11.16116736	94.52160494	0.790123457	506.25
10	28.52450069	94.52160494	0.790123457	6806.25
11	34.11533403	94.52160494	0.790123457	20306.25
12	13.38950069	94.52160494	2.595679012	1406.25
13	7.071167361	94.52160494	0.37345679	6806.25
14	0.116167361	94.52160494	0.37345679	506.25
15	0.707000694	94.52160494	0.37345679	6806.25
16	1.797834028	94.52160494	0.37345679	20306.25
17	11.16116736	94.52160494	5.706790123	1406.25
18	0.116167361	94.52160494	0.790123457	1406.25
Sum	153.5673458	9823.611111	17.27777778	77512.5
	S_y	S_{x_1}	S_{x_2}	S_{x_3}
	2.816	22.74	0.954	63.87

A new scale is obtained:

$$\bar{x}_j^0 = 0; \quad \bar{y}^0 = 0; \quad S_{xj}^0 = 1; \quad S_{y^0} = 1.$$

Indicators of conversion from natural scale to dimensionless scale are given in Table 4.

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Table 4

Conversion from natural scale to dimensionless scale

Experiment. №	$\frac{y_i - \bar{y}}{S_y}$	$x_{1i}^0 = \frac{x_{1i} - x_1}{S_{x_1}}$	$x_{2i}^0 = \frac{x_{2i} - x_2}{S_{x_2}}$	$x_{3i}^0 = \frac{x_{3i} - x_3}{S_{x_3}}$
1	–0.944154869	–2.65093118	0.640845057	–0.587113675
2	–0.890896368	–2.43103827	0.640845057	–0.587113675
3	–0.944154869	–1.33157372	0.640845057	–0.587113675
4	–0.944154869	–0.45200209	0.640845057	–0.587113675
5	–0.589098196	0.427569545	0.640845057	–0.587113675
6	–1.121683206	1.307141179	0.640845057	–0.587113675
7	0.506251639	0.427569545	–1.456466038	–0.587113675
8	–0.187884156	0.427569545	–0.407810491	–0.587113675
9	1.186185168	0.427569545	–0.932138264	0.352268205
10	1.896298513	0.427569545	–0.932138264	1.291650085
11	2.07382685	0.427569545	–0.932138264	2.231031965
12	–1.299211542	0.427569545	1.689500604	–0.587113675
13	–0.944154869	0.427569545	0.640845057	–1.291650085
14	0.121015149	0.427569545	0.640845057	0.352268205
15	0.298543486	0.427569545	0.640845057	1.291650085
16	0.476071822	0.427569545	0.640845057	2.231031965
17	1.186185168	0.427569545	–2.505121585	–0.587113675
18	0.121015149	0.427569545	–0.932138264	–0.587113675

Table 5

Calculation of selectivity coefficient of correlation

Experiment. №	y_{x1}	y_{x2}	y_{x3}
1	2.5029	–0.6051	–0.8368
2	2.1658	–0.5709	–0.7896
3	1.2572	–0.6051	–0.8368
4	0.4268	–0.6051	–0.8368
5	–0.2519	–0.3775	–0.5221
6	–1.4662	–0.7188	–0.9942
7	0.2165	–0.7373	0.4487
8	–0.0803	0.0766	–0.1665
9	0.5072	–1.1057	2.1656
10	0.8108	–1.7676	5.2434
11	0.8867	–1.9331	7.6824
12	–0.5555	–2.1950	–1.1515
13	–0.4037	–0.6051	–0.1716
14	0.0517	0.0776	0.2209
15	0.1276	0.1913	0.8255
16	0.2036	0.3051	1.7636
17	0.5072	–2.9715	1.0513
18	0.0517	–0.1128	0.1073
	r_{yx_1} =0.4093	r_{yx_2} =-0.8388	r_{yx_3} =0.7766

The selectivity coefficient of the correlation will be calculated by formula (7), (8):

$$r^*_{y^0 x_j^0} = \frac{1}{n-1}\sum_{i=1}^{n} y_i^0 x_{ji}^0 \quad , \tag{7}$$

$$r^*_{x_j^o x_m^o} = \frac{1}{n-1}\sum_{i=1}^{n} x_{1_i}^0 x_{m_i}^o , \qquad l, m = 1,2,3, l > m. \tag{8}$$

The values of sample pairwise correlation coefficients are depicted in Table 6.

Table 6

Sample pairwise correlation coefficients

Experiment. №	x_1^0	x_2^0	x_3^0	$x_1^0 \cdot x_2^0$	$x_1^0 \cdot x_3^0$	$x_2^0 \cdot x_3^0$
1	–2.65093	0.64085	0.88632	–1.69884	–2.34958	0.56800
2	–2.43104	0.64085	0.88632	–1.55792	–2.15469	0.56800
3	–1.33157	0.64085	0.88632	–0.85333	–1.18021	0.56800
4	–0.45200	0.64085	0.88632	–0.28966	–0.40062	0.56800

Continuation of Table 6

5	0.42757	0.64085	0.88632	0.27401	0.37897	0.56800
6	1.30714	0.64085	0.88632	0.83767	1.15855	0.56800
7	0.42757	–1.45647	0.88632	–0.62274	0.37897	–1.29090
8	0.42757	–0.40781	0.88632	–0.17437	0.37897	–0.36145
9	0.42757	–0.93214	1.82571	–0.39855	0.78062	–1.70181
10	0.42757	–0.93214	2.76509	–0.39855	1.18227	–2.57744
11	0.42757	–0.93214	3.70447	–0.39855	1.58392	–3.45308
12	0.42757	1.68950	0.88632	0.72238	0.37897	1.49745
13	0.42757	0.64085	0.18179	0.27401	0.07773	0.11650
14	0.42757	0.64085	1.82571	0.27401	0.78062	1.16999
15	0.42757	0.64085	2.76509	0.27401	1.18227	1.77199
16	0.42757	0.64085	3.70447	0.27401	1.58392	2.37399
17	0.42757	–2.50512	0.88632	–1.07111	0.37897	–2.22035
18	0.38152	–0.93214	0.88632	–0.35563	0.33815	–0.82618
				$r_{x_1^0x_2^0}$	$r_{x_1^0x_3^0}$	$r_{x_2^0x_3^0}$
				–0.25733	0.23567	–0.11017

The calculated correlation selectivity coefficient equals the correlation coefficient between variables expressed on a natural scale $r^*_{yx_j}$, $r^*_{x_lx_m}$. The normalized unter variate regression equation does not contain a complimentary term and takes the form (9):

$$\hat{y}^0 = a_1x_1^0 + a_2x_2^0 + a_3x_3^0 \quad (9)$$

The coefficients of the equation are found using the following relationship (10):

$$S = \sum_{i=1}^{n}(y_i^0 - \hat{y}_i^0)^2 = \min. \quad (10)$$

As a result of transformations, we get a system of standard equations (11):

$$\begin{aligned} &a_1 = a_2r^*_{x_1x_2} + a_3r^*_{x_1x_3} = r^*_{yx_1} \\ &a_1r^*_{x_2x_1} + a_2 + a_3r^*_{x_2x_3} = r^*_{yx_2} \\ &a_1r^*_{x_3x_1} + a_2r^*_{x_3x_2} + a_3 = r^*_{yx_3} \end{aligned} \quad (11)$$

Let's replace the calculated values of the correlation coefficient with a system of linear equations:

$$\begin{aligned} &a_1 - 0.2573a_2 + 0.2357a_3 = 0.4093, \\ &-0.2573a_1 + a_2 - 0.1102a_3 = -0.8388, \\ &0.2573a_1 - 0.1102a_2 + a_3 = 0.7766. \end{aligned}$$

We solve the system of linear equations and get the values of a_i coefficients:

$$a_1 = 0.0560; \quad a_2 = -0.7494; \quad a_3 = 0.6809.$$

Calculation of multiple correlation coefficient:

$$R = \sqrt{a_1r^*_{yx_1} + a_2r^*_{yx_2} + a_3r^*_{yx_3}} = 0.9562.$$

The correlation coefficient is close to unity, which indicates a close relationship between the predictors and the output parameter.

Using the following formula, we transform the dimensionless coefficient equation into a natural scale equation (12):

$$b_j = a_j\frac{S_y}{S_{xj}} \quad (12)$$

$$b_1 = a_1\frac{S_y}{S_{x_1}} = 0.0560\cdot\frac{2.816}{22.74} = 0.0069$$

$$b_2 = a_2\frac{S_y}{S_{x_2}} = 0.7494\cdot\frac{2.816}{0.954} = -2.2134$$

$$b_3 = a_3\frac{S_y}{S_{x_3}} = 0.6809\cdot\frac{2.816}{63.87} = 0.0300.$$

Let's find the free member using the formula below (13):

$$b_0 = \bar{y} - \sum_{i=1}^{n} b_j\bar{x}_j = 41.6591 + 4.086 = 45.745. \quad (13)$$

Thus we get the following equation (14):

$$\hat{y} = 45.745 + 0.0069x_1 - 2.2134x_2 + 0.0300x_3. \quad (14)$$

We check the adequacy of the resulting equation to the level of significance (p = 0.05) using the Fisher criterion.

In the absence of parallel experiments, it is possible to assess how appropriate it is to approximate the variance of the mean value s_y^2 by the adopted equation S_{res}^2.

According to the Fisher criterion (15):

$$F = \frac{S_y^2(f_1)}{S_{res}^2(f_2)}. \quad (15)$$

Based on the obtained regression equation, the Fisher criterion shows how often the resulting variance decreases compared to the average variance. If the F value exceeds the table value

$F_{1-p}(f_1, f_2)$ for the selected degree of significance p and level of freedom $f_1 = n - 1$ и $f_2 = n - l$, (l – is the number of coefficients in the equation), the regression equation is more effective.

The residual variance is defined by the following equation(16) and the calculated results are given in Table 7:

$$S_y^2 = \frac{\sum_{i=1}^{n}(y_i - \bar{y})^2}{n-1}, \quad (16)$$

s_y – variance is predetermined and is equal to 2.288.

Table 7

Values of residual variance

Experiment. №	y	x_1	x_2	x_3	$\hat{y}$	$(y_i - \hat{y}_i)^2$
1	39	10	4	60	38.76261694	0.056350716
2	39.15	15	4	60	38.79729631	0.124399892
3	39	40	4	60	38.97069316	0.000858891
4	39	60	4	60	39.10941064	0.011970688
5	40	80	4	60	39.24812812	0.565311325
6	38.5	100	4	60	39.3868456	0.786495116
7	43.45	80	2	60	43.67481926	0.050543702
8	40.26	80	3	60	41.46147369	1.443539032
9	45	80	2.5	120	44.36953941	0.397480554
10	47	80	2.5	180	46.17093234	0.687353177
11	47.5	80	2.5	240	47.97232528	0.223091168
12	38	80	5	60	37.03478255	0.931644732
13	39	80	4	15	37.89708342	1.216424984
14	42	80	4	120	41.04952105	0.903410229
15	42.5	80	4	180	42.85091399	0.123140626
16	43	80	4	240	44.65230692	2.730118156
17	45	80	1	60	45.88816484	0.788836777
18	42	80	2.5	60	42.56814648	0.32279042
			Total			11.36376018
			Residue S_{res}^2			0.710235012

$$S_{res}^2 = \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{n-1} \cdot F = \frac{S_y^2(f_1)}{S_{res}^2(f_2)} = \frac{2.816^2}{0.7102} = 11.1682$$

Finding the table value of Fisher's criterion:

$$F_{1-p}(f_1, f_2) = F_{1=0.05}(20-1,\ \ 20-4) = \mathrm{F}_{0.95}(17,\ 14) = 2.288$$
$$F_{1-p}(f_1, f_2) = F_{1-0.05}(20-1,\ 20-4) = F_{0.95}(17,\ 14) = 2.288$$

Attitude Assessment:

$$\frac{F}{F_{1-p}(f_1, f_2)} = \frac{11.1682}{2.288} = 4.880.$$

The calculated value of Fisher's criterion exceeds the table value by 4.88 times, indicating the regression equation's adequacy. For comparison, primary data analysis was carried out using the OriginLab program.

As can be seen from the presented results, the coefficients of the regression equation obtained for analysis in OriginLab coincide with the coefficients obtained during calculations in OptimMe [26].

Comparative characteristic dependences of benzene yield according to experiments and model are shown in Fig. 3.

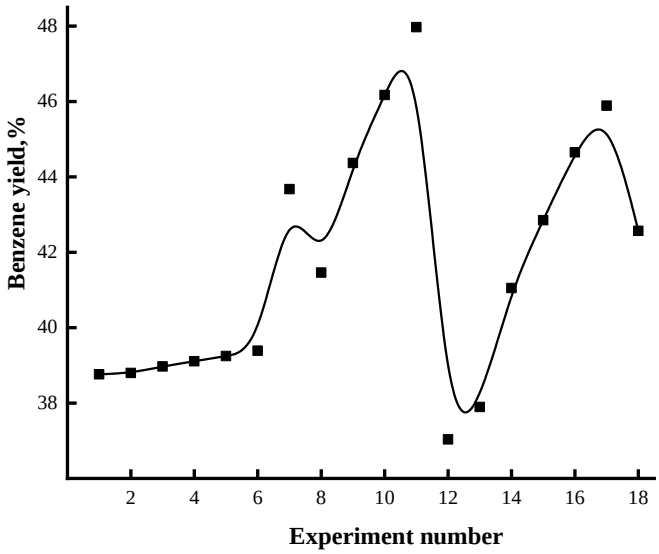

Fig. 3. Variation of benzene yield according to experiment number: points-experimental indicators, curve-model indicators

Thus, Fig. 3 shows that the dependencies of the experiment and the model of catalytic processing of light resin coincide, and therefore the model is valid. The model can be used for further practical work and allows one to calculate the benzene yield for any mode parameters.

Conclusion

1. An effective catalyst for the processing process was synthesized in the laboratory, and catalytic processing for light resin was carried out.

2. The optimal mode corresponding to the maximum yield of benzene in the system was determined, and high-purity benzene was obtained.

3. Calculations were made in a unique software package, regression equations of the process were found, and mathematical modeling was developed. The obtained regression equation allows us to calculate benzene yield at any catalyst concentration and the process's temperature regime.

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