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KINETICS AND MECHANISMS OF THE PROCESS OF GETTING VINYL ACETATE FROM ACETYLENE

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ABSTRACT

The essence of the process of synthesis of vinyl acetate from acetylene is the production of vinyl acetate from acetylene and acetic acid in the presence of zinc acetate catalyst in the vapor-gas phase in the static catalyst layer.Numerous studies on the synthesis of vinyl acetate in the vapor phase have been published, the process is carried out at atmospheric pressure in the range of 170-230 ° C, acetylene: acetic acid mole ratio in the presence of zinc acetate ingested on activated carbon. Replacement of zinc acetate with partial or complete cadmium acetate leads to an increase in catalyst activity. $K_2Cr_2O_7$ (2% relative to catalyst mass) is used as a promotional additive.Sensitive elements were installed to measure the temperature at three points along the height of the reactor and catalyst layer to measure the exothermic reaction heat. In the experiments, the ratios of the initial components were changed within the limits that provide low conversion of the given component in a stoichiometrically low amount.

KEYWORDS: *acetylene, acetic acid, vinyl acetate,*

INTRODUCTION

At present, in the production of vinyl acetate as a reaction apparatus are used mainly fixed-bed tubular reactors. The process of vapor phase catalytic synthesis of acetylene and acetic acid from acetylene and acetic acid in a coal zinc-acetate catalyst involves a number of additional reactions in addition to the main reaction, including the separation of reactions leading to the formation of acetaldehyde, acetone, croton aldehyde, water and carbon dioxide possible [1-7].

The essence of the process of synthesis of vinyl acetate from acetylene is the production of vinyl acetate from acetylene and acetic acid in the presence of zinc acetate catalyst in the vapor-gas phase in the static catalyst layer [8-14].

V is the reaction of formation of vinylacetate

$$C_2H_2 + CH_3COOH \longrightarrow CH_2 = CH - O - \underset{\underset{O}{\|}}{C} - CH_3 + 28\ \text{ккал}$$

Temperature (185 + 230°C)

The reaction of formation of additional products

In addition to the main reaction, the following additional reactions take place during the synthesis of vinyl acetate:

$$2CH_3COOH \rightarrow (CH_3)_2CO + H_2O + CO_2$$
$$C_2H_2 + H_2O \rightarrow CH_3CHO$$
$$2C_2H_2 + H_2O \rightarrow CH_2CH = CHCHO$$

Formation of acetalaldehyde and acetic acid due to hydrolysis of vinyl acetate:

$$CH_2 = CHOCOCH_3 + H_2O \longrightarrow CH_3\overset{\overset{O}{\|}}{C}\text{-}H + CH_3COOH$$

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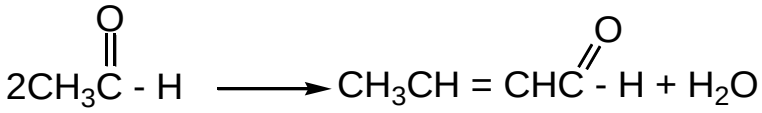
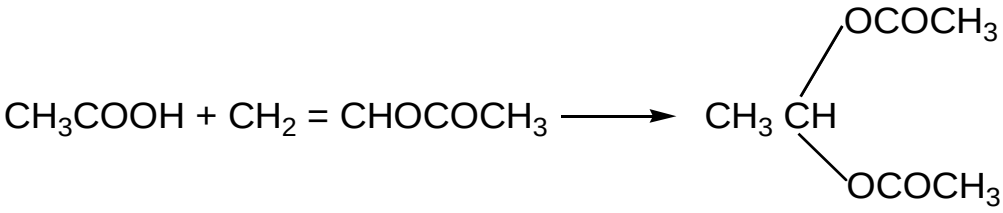
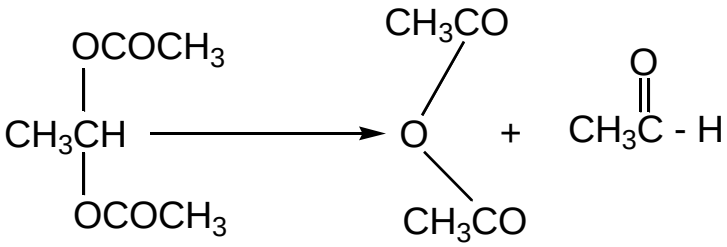
K roton aldehyde :



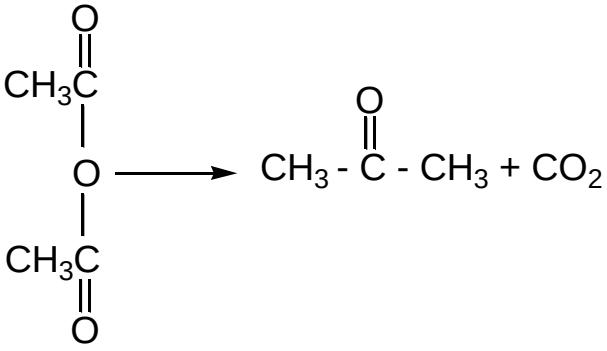
of Ethylidendiacetate :

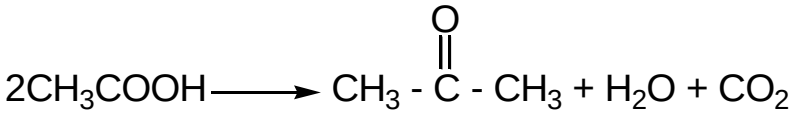


By obtaining acetic anhydride and acetaldehyde decomposition of ethylidendiacetate :

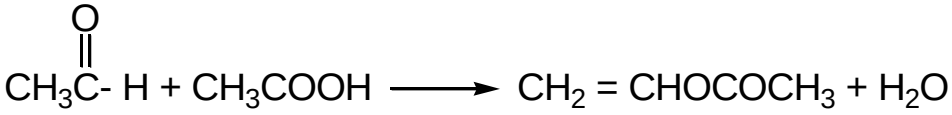


A cetone formation

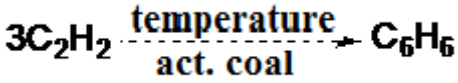




Acetylation of a cetaldehyde



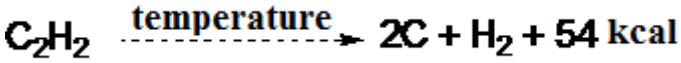
of Benzol



A cetaldehyde, croton aldehyde, vinyl acetate, acetylene and its homologues, the formation of adhesive products due to the polymerization of acetic anhydrides ;

The presence of copper (acetylene molecule, amorphous yellow substance, not immersed in water and insoluble in ordinary solutions) allows the presence of copper in acetylene cuprenization and contact with heated metal surfaces.In the formation of cuprenes - a decisive factor is the increase in the time of formation of the vapor-gas mixture in the contact zone ;

Decomposition of acetylene A :



is carried out in the presence of a large amount of acetylene .

Additional reactions may be caused by :

a) increase in moisture content of raw materials (acetylene, acetic acid ,catalyst);

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b) of the process wrong take to go or catalyst local heat up departure ;
c) acetylene andthe presence of compounds in acetic acid ;
C. Causes of reduced catalyst activity during synthesis ;
A) formation of viscous substances that close the pores of the catalyst and reduce their active centers, making it difficult to fly;
B) partial removal of zinc acetate from the reaction zone.
The inactivation kinetics of the zinc acetate catalyst under consideration has been studied in many studies. However, insufficient coverage of additional reactions in the developed models and insufficient description of the existing experimental data using them led to the need to set tasks for the development of kinetic models of the process of vinylacetate synthesis and catalyst activation [102].

EXPERIMENTAL PART

The reaction products were analyzed chromatographically: Tsvet-100 chromatography with flame ionization detector; glass column with size 2x0.04m, 15% lestosil in stationary phase Tsvetochrome (d = 0.250-0.315mm), column thermostat temperature 100 ^{o} C, evaporator temperature 120 ^{o} C, carrier gas (nitrogen) consumption 30 ml / min. Qualitative analysis was performed based on a comparison of 'witnesses' and capture sizes, using quantitative analysis and internal normalization methods. Data on the texture characteristics of the samples were obtained by low-temperature adsorption of liquid nitrogen at 77.35 K on an ASAP 2010 M instrument. Samples analysis to be done 4 hours before dried at 120 ^{0}C during and 6 hoursburned at 550 ^{0}C during . Comparison surface surface BET method detected . Of the poresgeneral size maximum when saturated adsorbed of nitrogen amount on was calculated .The distribution of pores by size was determined by the BJH (Barrett-Joyner-Halender) method.

Numerous studies on the synthesis of vinyl acetate in the vapor phase have been published [13; S. 973-976. 14 -15], the process is carried out at atmospheric pressure in the range of 170-230 ° C, acetylene: acetic acid mole ratio in the presence of zinc acetate ingested on activated carbon. Replacement of zinc acetate with partial or complete cadmium acetate leads to an increase in catalyst activity. $K_2Cr_2O_7$ (2% relative to catalyst mass) is used as a promotional additive.

EXPERIMENTAL RESULTS AND THEIR DISCUSSION

In the reaction of heterogeneous-catalytic acetylation of acetylene for the first time the catalytic activity of catalysts prepared from salts of d-elements prepared in the technology "Zol-gel" was studied. The results obtained are presented in Table 3.1. (Table 3.1).

Table 3.1

Influence of starting materials on the catalyst activity in the catalytic acetylation reaction of acetylene (T = 453 K, C_2N_2 : CH_3COOH 4: 1, V_{C2H2} = 280 h^{-1}, promoter: 1.8% $K_2Cr_2O_7$) [94]

№	Catalyst composition	Of CN_3COOH conversion ,%		Selectivity S %
		General	Vinyl acetate	
1	ZnO / keramzite	60.0	43.0	71.1
2	ZnO: CdO / keramzite	80.6	73.5	91.2
3	ZnO: ZrO_2 / keramzite	51.4	38.2	74.3
4	ZnO: CdO: ZrO_2 / keramzite	85.4	79.8	93.4
5	ZnO: Cr_2O_3 / keramzite	46.2	30.6	66.2
6	Cr_2O_3 : CdO: ZrO_2 / keramzite	67.8	49.2	72.5
7	ZnO: Cr_2O_3 : ZrO_2 / keramzite	72.1	51.9	72.0
8	ZnO: Fe_2O_3 : Cr_2O_3 : / keramzite	70.9	48.0	67.7

Under the selected normal conditions of the reaction, the total conversion of acetic acid is 85.4%, and the conversion to vinyl acetate is 79.8%.

V The influence of various factors (temperature, molecular ratios of starting materials, volumetric velocity, the effect of reaction products, etc.) on the rate of the process of inylacetate synthesis was studied.

The effect of temperature rise rate on catalyst activity. When the temperature rises rapidly to 180 ° C, the formation of resins and polymers on the surface of the catalyst decreases and the activity of the catalyst increases. The results obtained are shown in Figure 3.1.

As can be seen from Figure 3.1, when the temperature rises rapidly to 180 ^{o} C for 30 days, the yield of vinyl acetate increases by 2–3 times per 12 cups as the temperature rises slowly, and the total yield for the whole synthesis increases by 15–20%.

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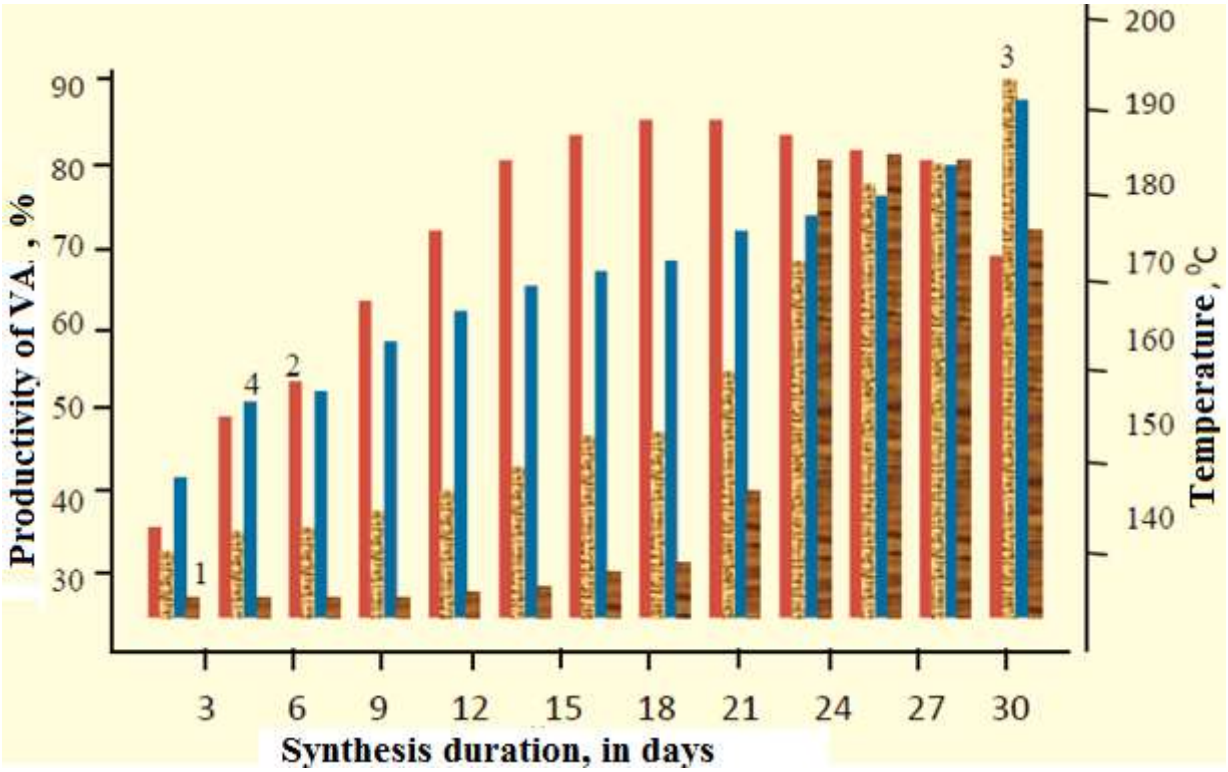

1 and 2 vinyl acetate yields; Temperatures in reactors 3 and 4

Figure 3.1. The dependence of the catalyst activity on the rate of temperature rise

C_2H_2 : CH_3COOH molar ratio. It is of great interest to study the effect of the ratio of C_2H_2 : CH_3COOH on the catalyst yield during the synthesis of vinyl acetate .

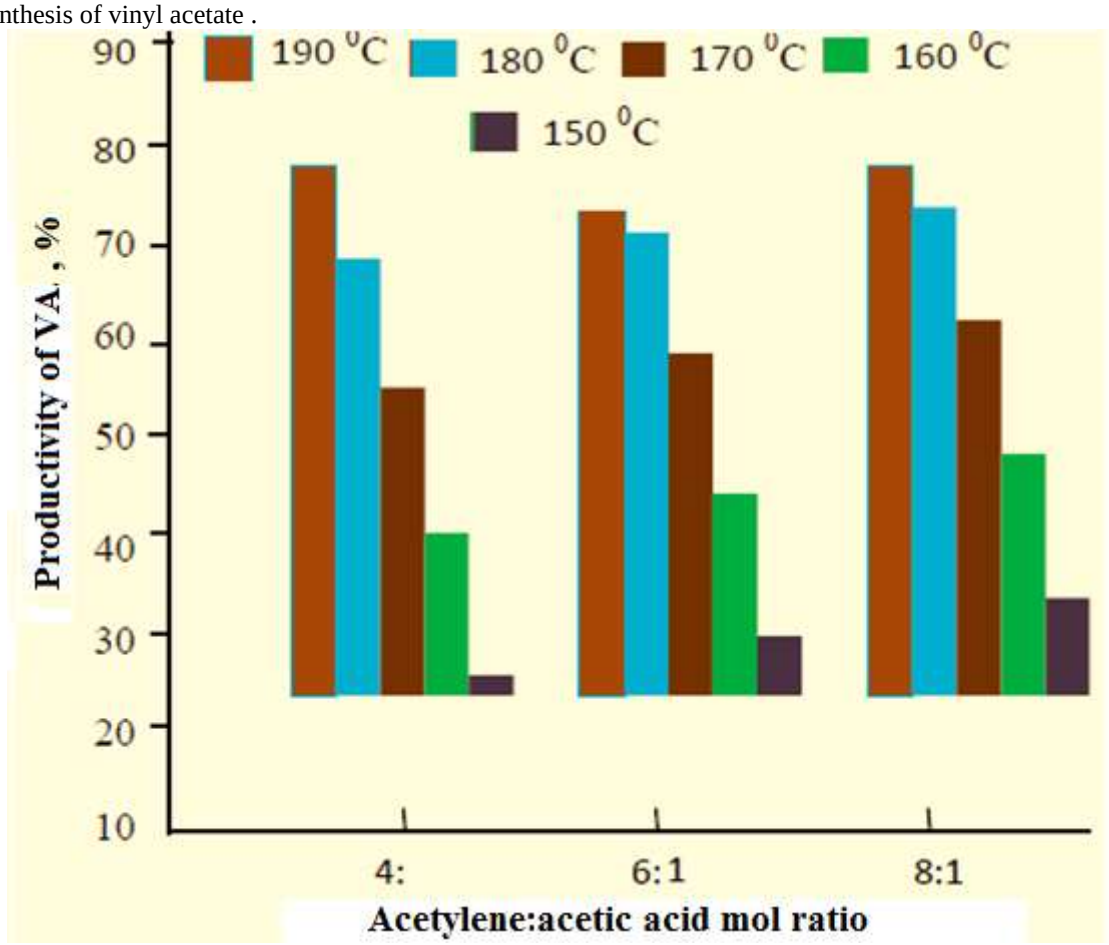

Figure 3.2. Acetylene: the effect of acetic acid mole ratio on vinyl acetate yield

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For this purpose, at 150 ^{o} C, 160 ^{o} C, 170 ° C and 180 ° C, the molar ratio of C_2H_2 : CH_3COOHs is 4: 1 to 8 at a constant volumetric velocity of 270 g / l · cat · h of the vapor-gas mixture. : We conducted experiments by changing to 1. The results obtained are shown in Figure 3.2.

As can be seen from Figure 3.2, the activity of the catalyst increases to 180 ° C as the amount of acetylene increases . When the temperature exceeds 185 ° C, the reaction yield decreases due to the decomposition of the vinyl acetate and the formation of additives. Hence, the rate of the reaction depends on the amount of acetylene and not on the amount of acetic acid.

The dependence of the volumetric velocity of the vapor-gas mixture on the catalyst efficiency. It is known that the efficiency of a catalyst depends on the normal volumetric velocity of the vapor-gas mixture. To find the optimal volumetric velocity of the vapor-gas mixture, we carried out the synthesis under conditions of constant molar ratio C_2H_2 : CH_3COOH = 1: 6 and at different values of the volumetric velocity. The dependence of the volumetric velocity on the catalyst efficiency at different temperatures is shown in Figure 3.3.

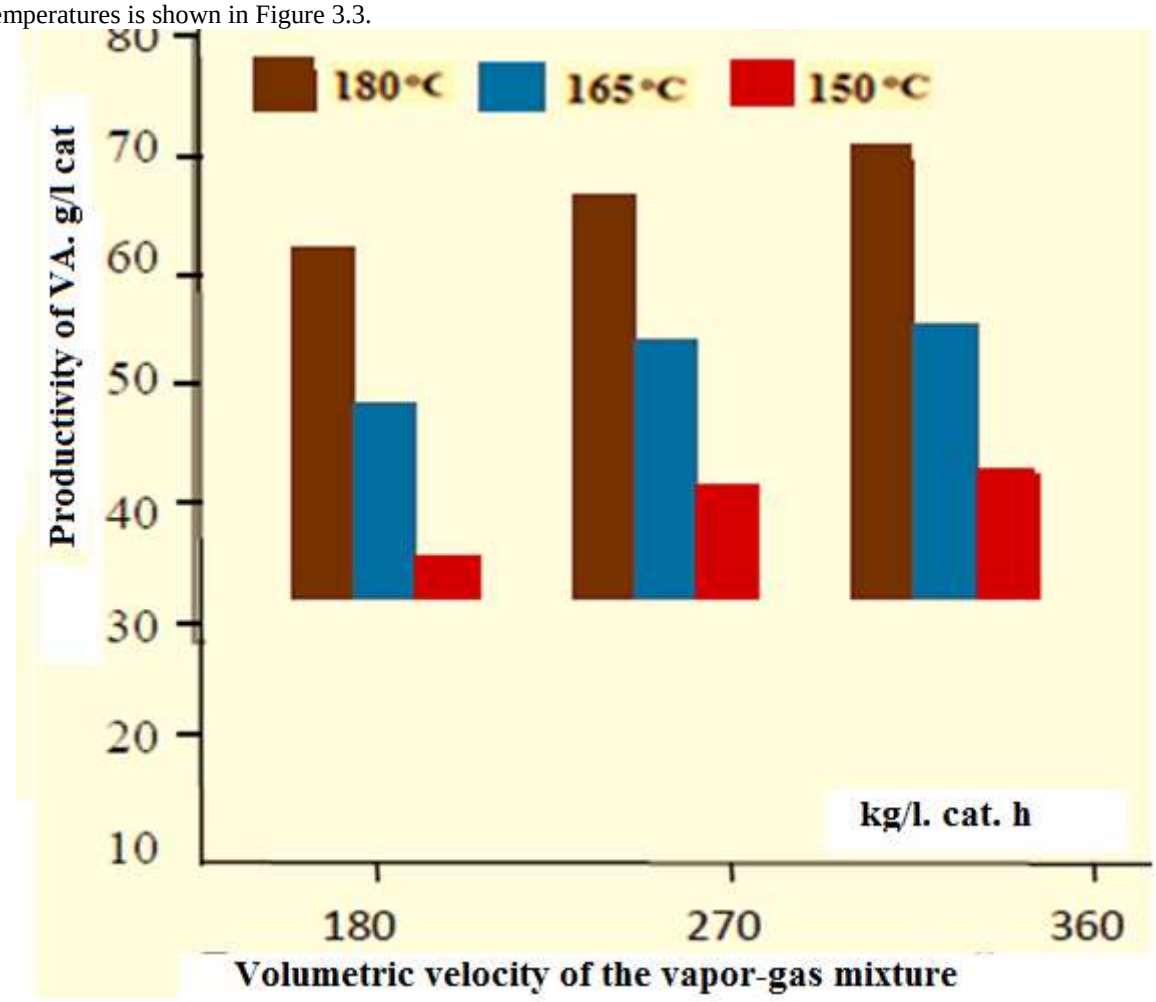

Figure 3.3. Acetylene: the effect of the volumetric velocity of a mixture of acetic acid on the yield of vinyl acetate

As can be seen from Figure 3.3, the efficiency of the catalyst increases by 10-15% when the volumetric velocity of the acetylene: acetic acid mixture is increased from 180 to 360 kg / l · kat · h. Further increase in volumetric velocity is not effective. This is because at this time the conversion of acetic acid is reduced and the vinyl acetate contains 65-70% acetic acid. This complicates the rectification process.

The activity of the catalyst and the service life of the catalyst depend on the nature and quality of the core substance (carrier). Of all the core substances (carriers) we have tested (silica gel, alumina, expanded clay, activated carbon of various brands), the most active is expanded clay. Therefore, in this study , the results obtained in the presence of a catalyst consisting of a mixture of expanded clay $(ZnO)_{0.5} \cdot (CdO)_{0.45} \cdot (ZrO_2)_{0.05}$/ expanded clay were described.

100 grams of catalyst was placed in the reactor, and in some experiments 150-200 grams. The size of the catalyst particles is 0.20-0.25 mm.

To study the effect of the catalyst composition on the catalytic activity of a mixture of zinc and cadmium acetates, we changed the amount of zinc acetate and cadmium acetate in the catalyst from 0 to 30%. We examined the catalytic activity of each sample at 150 ° C , 170 ° C , and 1 8 0 ° C. The results are shown in Figure 3.4.

From the data shown in Figure 3.4, it can be seen that the cadmium acetate catalyst and the zinc acetate catalyst have relatively less catalytic activity when used separately. However, a catalyst consisting of a mixture of zinc acetate and cadmium acetate exhibits high catalytic activity. As the concentration (amount) of cadmium acetate in the catalyst increases (17%), its

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catalytic activity increases and reaches a maximum, and then the catalytic activity decreases. Thus, the catalyst with the highest catalytic activity is a catalyst with a mass ratio of zinc acetate and cadmium acetate 1: 1.

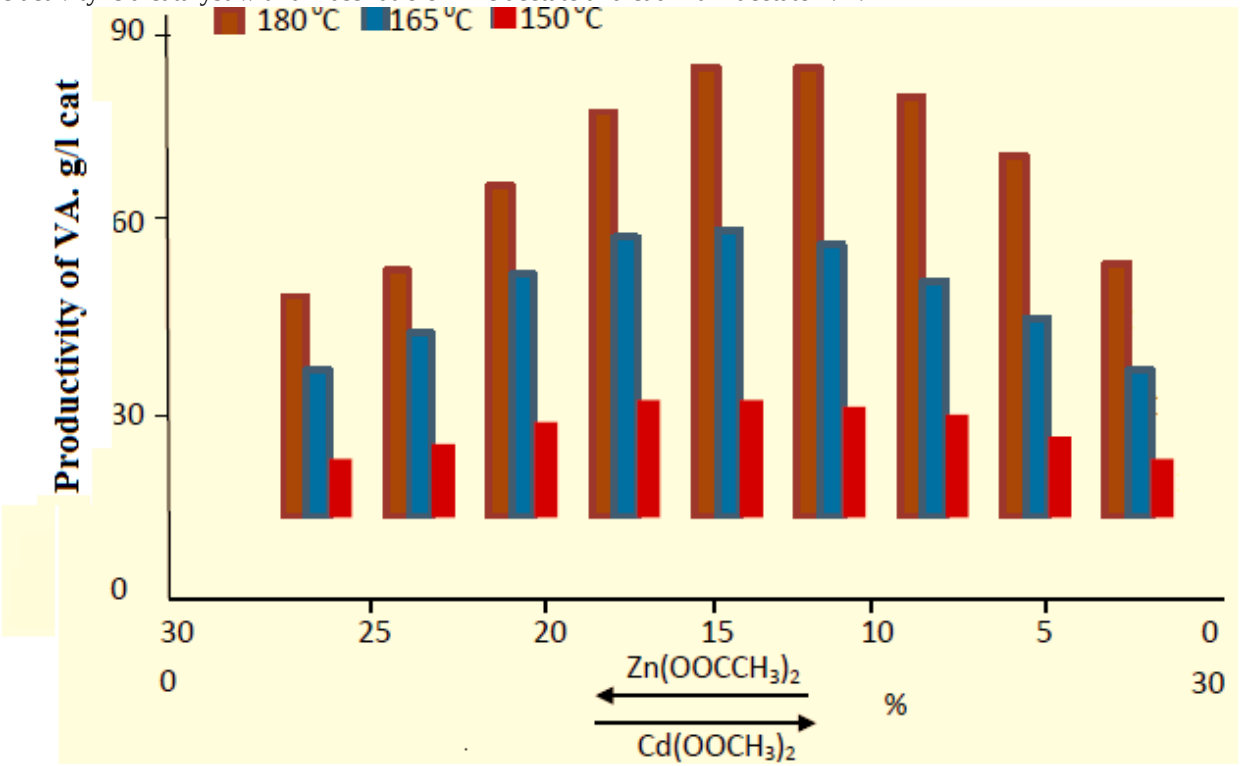

Figure 3.4. Dependence of catalyst activity on the ratio of zinc acetate and cadmium acetate when the total amount of acetates in it is 30%

The amount of vinyl acetate obtained is 25, 52, 94 g / l at 150 ° C, 170 ° C and 18 ° C. This is 1.6-1.7 times more than the amount of vinyl acetate obtained in the presence of zinc acetate catalyst.

The dependence of catalyst activity on zinc acetate and cadmium acetate amounts was studied. To do this, the total amount of zinc acetate and cadmium acetate was changed in the range of 10% to 40%, keeping the ratios of Zn $(CH_3COO)_2$ and Cd $(CH_3COO)_2$ constant at a 1: 1 ratio. The experiments were performed at 180 ° C and at a ratio of C_2H_2 : CH_3COOH = 4: 1. The results obtained are shown in Figure 3.5.

From the data shown in Figure 3.5, it can be seen that the yield of vinyl acetate increases when the sum of the concentrations of zinc acetate and cadmium acetate in the catalyst increases to 30-35%. When the total (total) amount of zinc acetate and cadmium acetate in the catalyst increases from 35 to 40%, the yield of vinyl acetate decreases.

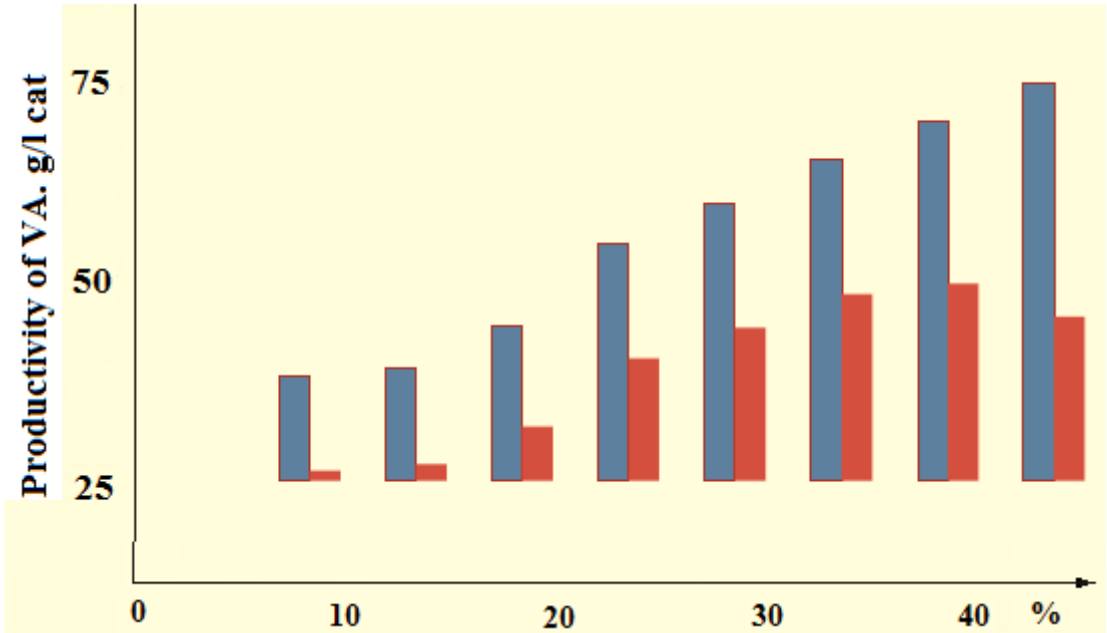

Figure 3.5. The dependence of the activity of the catalyst on the total amount of acetates in its composition

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Kinetic model of the process. Analysis of the data in the experiment and the literature allows us to express the following stoichiometric equations for the directions of change of the initial components to the main and by-products of the reaction [102]:

$S_2N_2 + SN_3SOON \overset{k_1}{\Rightarrow} SN_3SOOSNSN_2$

$W_1 = k_1 P^{n_1}_{CH3COOH} P^{n_2}_{C2H2} SN_3SOOSNSN_2 + N_2O \overset{k_2}{\Rightarrow} SN_3SNO + SN_3SOON$

$W_2 = k_2 P^{n_3}_{CH3COOH}$

$2SN_3SOON \overset{k_3}{\Rightarrow} SN_3SO\,SN_3 + N_2O + SO_2$

$W_3 = k_3 P^{n_3}_{CH3COOH} 2SN_3SNO \overset{k_4}{\Rightarrow} SN_3SNSNSNO + N_2O$

$W_4 = k_4 P^{n_4}_{CH3CHO}$

where $R_{SN3SOON}$, R_{S2N2}, R_{SN3SNO} - partial pressures of acetic acid, acetylene and acetaldehyde in the vapor-gas mixture, respectively; n_1 - n_4 - reactions procedures; W_1 - W_4 - reaction rates in the corresponding directions; k_1 - k_4 - constants of reaction rates.

Sensitive elements were installed to measure the temperature at three points along the height of the reactor and catalyst layer to measure the exothermic reaction heat. In the experiments, the ratios of the initial components were changed within the limits that provide low conversion of the given component in a stoichiometrically low amount. This allows the current reactor used in the first approach to be considered as an isothermal and differential reactor. Experimental data on the different values of the molar ratios of acetylene to acetic acid in the new catalyst, the volumetric velocity of the vapor-gas mixture, and the synthesis temperature are presented in Tables 4.1-4.3, respectively.

Table 4.1

T = 453 °K, the dependence of the acetylene molar ratio of the vapor-gas mixture on the acetic acid mole ratio at the exit of the reactor at a volumetric velocity of 200l / l (cat) hour

Mol ratio	Component composition , mol											
	Acetylene		Acetic acid		Vinyl - acetate x10		Acetal-degid x10 ^{3}		Acetone x10 ^{3}		Croton aldehyde x10 ^{4}	
	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal
2: 1	0.6 5	0.63	0.32	0.33	0.29	0.32	0.2 1	0.23	0.22	0.24	0.59	0.63
3: 1	0.7 5	0.74	0.22	0.23	0.2 5	0.2 6	0.1 8	0.1 6	0.18	0.1 7	0.46	0.4 4
4: 1	0.77	0, 80	0.20	0.18	0.24	0.2 2	0.14	0.1 3	0.1 5	0.13	izi	0.34
5: 1	0, 80	0.81	0.18	0.1 7	0.2 1	0, 20	0.1 3	0.11	0.1 1	0.1 2	izi	0, 30

Table 4. 2

T = 453 °K and at a ratio of 3: 1 mol at the exit of the reactor

V vol.frequency	Component composition , mol											
	Acetylene		Acetic acid		Vinilacetate x 10		Acetaldehyde x 10 ^{3}		Acetone x10 ^{3}		Croton aldehyde x10 ^{4}	
	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal
900	0.76	0.74	0.2 1	0.22	0.3 1	0.3 4	0.1 6	0.2 1	0.2 1	0.22	0.88	0.78
1200	0.7 5	0.74	0.22	0.23	0.2 5	0.25	0.1 8	0.1 6	0.18	0.1 7	0.46	0.4 4
1500	0.74	0.25	0.2 5	0.23	0.22	0.20	0.1 4	0.1 3	0.12	0.1 4	izl	0.28

Table 4.3

Volumetric velocity 1500 l / l (cat) hour and 5: 1 mol Dependence of temperature (T, K) on the composition of the vapor-gas mixture at the exit from the reactor

T, K	Component composition , mol											
	Acetylene		Vinegar is a lot		Vinilacetate x10		Acetaldehyde x10 ^{3}		Acetone x10^3		Croton aldegid x10 ^{4}	
	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal	exp	cal
443	0.84	0.83	0.14	0.16	0.07	0.0 8	0.02	0.0 3	0.0 3	0.0 3	izl	0.05
453	0.84	0.83	0.14	0.15	0.14	0.15	0.09	0.0 9	0.95	0.09	izl	0.19
463	0.81	0.8 3	0.1 6	0.14	0.2 6	0.2 8	0.27	0.25	0.2 6	0.2 7	izl	0.6 4
473	0.8 2	0.8 3	0.1 3	0.1 3	0.45	0.48	0.7 4	0.7 1	0.7 1	0.7 5	26	2.0 7
483	0.8 1	0.8 2	0.11	0.1	0.75	0, 80	1, 70	1.82	7.82	1.94	6.9	5.97

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Inverse of the relative errors were used to measure the concentrations of the respective components as weight coefficients. Because the calculated value of the Pekle parameter for the process under consideration is large enough (more than 250), which indicates that the longitudinal mixing effects cannot be bypassed, the hydrodynamic regime in the tube corresponds to the ideal compression mode and the compression model was chosen for its mathematical description. The following values of the constants are obtained:

$$n_1 = 0.24;\ n_2 = 0.76;\ n_3 = 1.0;\ n_4 = 1.0;$$
$$K_1 = 3{,}811 \cdot 10^{-5} \exp(-1282.0\ (1 / T\text{-}1/483));$$
$$K_2 = 0.1421 \cdot 10^{-5} \exp(-23371.3\ (1 / T\text{-}1/483));$$
$$K_3 = 0.0024 \cdot 10^{-5} \exp(-20102.2\ (1 / T\text{-}1/483));$$
$$K_4 = 0.7499 \cdot 10^{-5} \exp(-2085.6\ (1 / T\text{-}1/483));$$

In the development of mathematical models of the process of production of vinyl acetate from acetylene, an analysis of the impact of one or another hypothesis on the accuracy of the description of the modeled processes was conducted. The process of obtaining vinyl acetate from acetylene in the presence of acetic acid also made it possible to simplify the initial conditions for solving the problem of modeling the motion of technological elements. The program modules were implemented by assigning appropriate modes of operation modes (static modes calculation, static modes calculation, dynamic modes calculation, type of incoming excitations).

The analysis of the description errors showed that for all the models listed below, the errors are within the range of experimental errors.

The calculated values of the concentrations of the components of the reaction gases at the outlet of the reactor and the relevant experimental data are given in Tables 4.1-4.3. The average errors of the description are 5.7% for the main products and 8.2% for the additional products, respectively, comparable to the experimental error rate and allow the proposed kinetic model to be considered adequate.

The rate of the vinyl acetate synthesis reaction from acetylene and acetic acid increases with increasing temperature regardless of the composition of the catalyst. This is also clear from the value of the temperature coefficient of the reaction rate given in Table 3.5.

Table 3.5
Temperature dependence of the reaction rate

Temperature, °C	**VA yield , g / l · hours**	**Reaction rate constant, g · mol / l · sec . K · 10 $^{-4}$**	**Temperature coefficient of the reaction**	**Average value**
170	108	3.48		1.41
180	158	5.10	1.46	
190	210	6.76	1.33	
200	312	10.10	1.36	
210	420	13.60	1.36	
220	645	20.70	1.52	

The fact that the average value of the temperature coefficient of the vinyl acetate formation reaction is not very large is the result of the multi-stage process.

To calculate the activation energy

$$E_a = -\frac{R \cdot \ln\left(\frac{k_{T_1}}{k_{T_2}}\right) \cdot T_1 \cdot T_2}{(T_2 - T_1)}$$

formula was used.

CONCLUSION

The essence of the process of synthesis of vinyl acetate from acetylene is the production of vinyl acetate from acetylene and acetic acid in the presence of zinc acetate catalyst in the vapor-gas phase in the static catalyst layer.Numerous studies on the synthesis of vinyl acetate in the vapor phase have been published, the process is carried out at atmospheric pressure in the range of 170-230 ° C, acetylene: acetic acid mole ratio in the presence of zinc acetate ingested on activated carbon. Replacement of zinc acetate with partial or complete cadmium acetate leads to an increase in catalyst activity. $K_2Cr_2O_7$ (2% relative to catalyst mass) is used as a promotional additive.Sensitive elements were installed to measure the temperature at three points along the height of the reactor and catalyst layer to measure the exothermic reaction heat. In the experiments, the ratios of the initial components were changed within the limits that provide low conversion of the given component in a stoichiometrically low amount.

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