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Optimization of Vinyl Acetate Synthesis Process

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Abstract

In this paper, we consider the texture characteristics of the developed highly active catalyst $(ZnO)_x$ $(CdO)_y$ $(ZrO_2)_z$ for the catalytic acetylation of acetylene based on the Sol-Gel technology, and also study the effect of some parameters (temperature, volumetric rate, C_2H_2 molar ratio : CH_3COOH, catalyst preparation procedure) for the course of this process. Based on the results obtained, a kinetic model is created that satisfies the synthesis of vinyl acetate. Taking into account the catalyst deactivation, a mathematical model of the displacement reactor and determination of the main parameters of the tubular reactor was developed for the synthesis of vinyl acetate from acetylene and acetic acid. Using the developed nano-catalyst, the kinetic laws of vinyl acetate synthesis were studied, the material balance of the process was calculated, and an improved technological scheme for the synthesis of vinyl acetate was proposed. On the basis of stored information, vinyl acetate synthesis process has been optimized.

Keywords: *acetylene, acetic acid, catalyst, sol-gel technology, optimization process, product yield, process temperature, acetylene space velocity, catalyst bed height.*

1. Introduction

Vinyl acetate is widely used in industry mainly as a monomer. One of the important properties of vinyl acetate is its polymerization property. Of the polymer products obtained by vinyl acetate, polyvinyl acetate, polyvinyl alcohol and polyvinyl acetals are widely used. The fact that polyvinyl acetate has high adhesion and elasticity testifies to its high adhesive ability. Therefore, it is widely used in the production of water-soluble latex paints, adhesives, fibers, etc.

Today, the bulk of vinyl acetate is obtained from ethylene through its oxidative combination with acetic acid on gold-palladium catalysts, but the alternative method of producing vinyl acetate from acetic acid and acetylene also remains important. [1-3]:

1). Oxidative esterification of ethylene (Moses reaction). The process proceeds based on the reaction between ethylene, acetic acid and oxygen with the participation of a catalyst [4]:

$$C_2H_4 + CH_3COOH + ½O_2 \rightarrow CH_2=CHOCOCH_3 + H_2O$$

2). Based on steam-catalytic reaction between acetylene and acetic acid

$$C_2H_2 + CH_3COOH \rightarrow CH_2=CHOCOCH_3$$

Due to the low cost of ethylene compared to acetylene, the first method is more common. At present, cheaper sources of acetylene production are being found as an additional product of new production processes.

Therefore, the production process of vinyl acetate from acetylene remains relevant.

Today, zinc acetate deposited on crushed natural activated carbons of the ARD, ARD-U brands in Russia and S-76 abroad is used as a catalyst for the production of vinyl acetate from acetylene in reactors with a flowing bed. All of them retain the properties and disadvantages inherent in conventional activated carbons: they have high ash content, differ in an unbalanced ratio of the volumes of micro- and mesopores, which determines both a relatively low level of catalyst activity and its rapid deactivation.In addition, when using a reactor with a gushing layer, the low strength of the carrier and the irregular shape

of the particles lead to rapid abrasion of the catalyst and its entrainment from the reactor. A significant increase in the productivity of the synthesis process of vinyl acetate can be achieved by switching to zinc acetate catalysts obtained using nanoporous carbon microspheres as carriers, which are characterized by high strength, low abrasion resistance, and an optimal porous structure [9-17].

Based on the foregoing, the production of vinyl acetate is one of the main tasks of creating new, economic, waste-free methods in technology, as well as the development of effective catalysts with high selectivity, activity and productivity of modeling technological and operating parameters of the process [18-25].

2. Experimental part

The catalytic acetylene acetylation reaction was carried out in a flow reactor under the following optimal conditions: t = 180°C, C_2H_2: CH_3COOH = 4:1, Vc_{2H_2} = 280 h^{-1} [18, 24-25].

The reaction products were analyzed by a flame ionization detector by gas-liquid chromatography under the following optimal conditions: a stationary liquid phase with particle sizes of 0.250-0.315 mm in color chrome - 545, 15% lestosile, glass column 100°C, the flow rate of the incoming gas - nitrogen 30 ml/min.

Qualitative analysis of "witnesses" and the retention time of parameter values are based on a comparison method; and quantitative analysis is calculated based on the method of internal normalization [24-25].

Data on the texture characteristics of the samples were obtained on an ASAP 2010M instrument in a stream of liquid nitrogen at 77.35K by low-temperature adsorption. Before analysis, the samples were dried at 120°C for 4 hours and burned at 550°C for 6 hours. The comparable surface was determined by the BET method. The total surface volume was calculated based on the amount of nitrogen adsorbed at maximum saturation. Sponge size distributions were determined by the BJH method.

The phase composition was studied on a DRON-3 diffractometer (CuK_α radiation) by X-ray diffraction. The dispersion properties of the catalyst were checked in a scanning electron microscope (JSM - 6510 LV). The catalytic activity of the obtained sample was studied in acetylene acetylation reaction [27].

3. Experimental results and their discussion

The catalysts were prepared under the following conditions: solutions of a 5–25% solution of zirconium oxy nitrate by circular adsorption at 600 °C were absorbed onto a microsphericalnanoporous retention agent (expanded clay). The salt absorption time varied within 60-90 minutes.

The substance is the carrier expanded clay: the solution was taken in the range of ratios 1:3 - 1:8.5. After the absorption process, the catalyst was dried at room temperature for 24 hours, then in an oven at 100 - 130°C (with a temperature increase of 10°C every 1 hour). The amount of zinc acetate in the catalyst was 11-30%.

The catalyst, the volume of which was 9 cm^3, was lowered into a flow reactor and the system was washed in a stream of nitrogen at a speed of 10 l/h for 15 minutes. The synthesis of vinyl acetate from acetylene was carried out at normal atmospheric pressure at 180°C. Under the above conditions, the life of the catalyst was 2000 hours.

The synthesis of vinyl acetate in the vapor phase has been published in many works, the absorption process is carried out on activated carbon in the presence of zinc acetate at 170-230°C, the process was carried out at atmospheric pressure in the range of the molar ratio of acetylene: acetic acid from 2:1 to 10:1. Partial or complete replacement of zinc acetate with cadmium acetate leads to an increase in catalyst activity. $K_2Cr_2O_7$ (2% compared with the weight of the catalyst) is used as a promoter additive. For the first time in the catalytic acetylation reaction in the vapor phase of acetylene, we studied the catalytic activity of catalysts made from salts of d - elements obtained by the sol-gel method (Table 1).

Table 1. The Effect of Primary Substances on the Activity of the Catalyst in the Catalytic Acetylation of Acetylene.

(T = 453K, C_2H_2: CH_3COOH = 4:1, V c_{2H_2} = 280 hr^{-1}, promoter: 1.8% $K_2Cr_2O_7$)

№	Catalyst Structure	Conversion CH_3COOH, %		Selectivity S %
		General	According to 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

As can be seen from table 1, the catalyst (No. 4) containing oxides of zinc, cadmium, zirconium has a high yield and selectivity.

The table shows that the total conversion of acetic acid is 95.4%, with respect to the conversion of vinyl acetate is 79.8%.

With the participation of catalyst No. 4, we studied the yield of vinyl acetate, the selectivity of the process, and the conversion of the starting material under the influence of various factors (temperature, space velocity, molar ratios C_2H_2:CH_3COOH, catalyst preparation method, etc.).

When studying the effect of the ratios C_2H_2:CH_3COOH on the yield of vinyl acetate, as well as on the selectivity of the process, it was found that the optimal condition is the ratio C_2H_2: CH_3COOH 4:1.

Table 2. Effect of C_2H_2:CH_3COOH Ratios on Vinyl Acetate Yield (T = 180°C, catalyst No. 4)

Mole ratio C_2H_2:CH_3COOH	**Conversion of acetic acid,%**		**Selectivity, S %**
	General	**Vinyl acetate**	
1:3	**48.0**	**18.4**	**38.3**
1:2	**63.4**	**48.5**	**76.5**
1:1	**78.8**	**63.2**	**80.2**
2:1	**82.0**	**70.7**	**86.2**
3:1	**83.8**	**75.4**	**90.0**
4:1	**85.4**	**79.8**	**93.4**
5:1	**92.5**	**72.0**	**77.8**
6:1	**96.2**	**65.4**	**68.0**

As can be seen from the table, with an increase in the number of moles of acetylene in the reaction mixture, the yield of conversion of acetic acid increases.With an increase in the molar ratio of the output substances above 4: 1, the yield of vinyl acetate decreases, which is associated with the formation of a by-product (ethylene diacetate) In the temperature range 150-240°C with the participation of catalyst No. 4, the reaction of acetic acid vinylation (C_2H_2: CH_3COOH 4:1, $V_{C_2H_2} = 280\ h^{-1}$,cat NH.

Table 3. The Effect of Temperature on the Output Acetylene Acetylation Reaction

(T=T = 180°C, C_2H_2: CH_3COOH = 4:1, = 280 h^{-1} cat. No. 4)

	Temperature, °C	Conversion CH_3COOH,%	Selectivity

№				S %
		General	According to vinyl acetate	
1	150	58.4	40.5	69.3
2	165	72.5	59.8	82.5
3	180	85.4	79.8	93.4
4	195	90.2	78.4	86.9
5	210	92.8	73.5	79.2
6	225	94.9	65.4	68.9
7	240	96.8	55.8	57.6

When studying the effect of the temperature of the reaction of acetic acid vinylation within 150-240°C, an increase in the yield of vinyl acetate was observed with an increase in temperature to 185°C, and when the temperature was raised above 185°C, a decrease in the reaction yield was observed due to decomposition of vinyl acetate and the formation of additional substances.

In order to study the mechanism and kinetics of the catalytic acetylation reaction of acetylene on selected catalysts, we studied the influence of partial pressures of the starting materials over a wide range. The experiments were carried out at a constant gas flow rate, which was achieved by the addition of inert argon gas. As a result of studies, it was found that with an increase in the proportion of acetic acid and a decrease in the partial pressure of acetylene, the yield of vinyl acetate decreases.

The experiments were carried out with a change in a wide range of parameters that ensured the reaction in the kinetic region: temperature, partial pressure of the reagents, and specific speed of acetic acid. The influence of the partial pressures of the starting and final substances on the kinetic laws of acetylene acetylation was studied under the condition that the partial pressure of one component changes with the partial pressures of the remaining components being constant. To maintain a constant feed rate of the starting materials, if necessary, an inert gas (nitrogen) is supplied to the reaction zone.

It is proved that within the parameters in which the process is studied, the yield of vinyl acetate increases with increasing acetylene concentration and decreases with increasing partial pressure of acetic acid. Adding vinyl acetate to the reaction medium does not affect the rate of its formation. At temperatures above 210°C, the addition of water enhances the hydrolysis of vinyl acetate, but does not change the activity of the catalyst. With a molar ratio of acetylene: water = 10:1, a synthesis temperature of 200°C and a contact time of 4 seconds, the conversion of acetic acid reaches 100%. With the same ratio of components (10:1) and maintaining a temperature of 200°C, a reduction in contact time from 4 to 2 seconds yields vinyl acetate reaches 95-96%.

Based on the study, the influence of various factors on the reaction rate involving the selected $(ZnO)_x \cdot (CdO)_y \cdot (ZrO_2)_z$ / expanded clay containing catalyst, the kinetic laws of the process were studied, and a reaction mechanism was proposed. Based on experiments and chromatographic analyzes, as well as published data, the following reaction mechanism is proposed:

$$I.CH_3COOH + Z \rightarrow CH_3COOH * Z$$
$$II.CH \equiv CH + Z \rightarrow CH \equiv CH * Z$$
$$III.CH_3COOH * Z \rightarrow CH_3COO^- * Z^+H^+$$
$$IV.CH \equiv CH * Z + H^+ \rightarrow CH_2 = CH^+ * Z$$
$$V.CH_2 = CH^+ * Z + CH_3COOH \rightarrow CH_2 = CHOCOCH_3 * Z + H^+$$
$$VI.CH_2 = CHOCOCH_3 * Z \rightarrow CH_2 = CHOCOCH_3 + Z$$

– vinyl acetate

$$VII.CH_2 = CH^+ * Z + CH_3COOH * Z \rightarrow CH_2 = CHOH * Z + CH_3C^-O * Z$$
$$VIII.CH_2 = CHOH * Z \rightarrow CH_3CHO + Z$$

– – – – – – – – – – – – – – – acetaldehyde

$$IX.CH_3C^-O * Z + CH_3CHO \rightarrow CH_3OCOCH(CH_3)O^- * Z$$
$$X.CH_3OCOCH(CH_3)O^- * Z + CH_3C^+O * Z \rightarrow CH_3CH(OCOCH_3) + 2Z$$

– ethylidenediacetate

Z-active center of the catalyst. The reaction mechanism proposed above confirms and complements the theories available in the scientific literature. From the mechanism it is seen that ethylidenediacetate is not formed from vinyl acetate. With an increase in the partial pressure of acetic acid, the rate of formation of acetaldehyde increases. This, in turn, leads to an increase in the yield of ethylidenediacetate.

To interpret the obtained experimental data, it is necessary to find kinetic equations that satisfy a wide range of parameter changes (reaction rates, reaction rate constants, adsorption coefficient, and partial pressure). After determining the kinetic parameters of these equations, one can decide which one satisfies the experimental data. The detected kinetic constants and adsorption coefficients were used to determine the rate of synthesis of vinyl acetate from acetylene using various kinetic equations. Investigation of the influence of partial pressures of the starting material, space velocity and temperature. At the acetylene acetylation reaction rate, as well as the results of chromatographic analysis of the acetylation reaction, the following equation is satisfied:

$$W=K P_{C_2H_2} \cdot P_{CH_3COOH} /(1+b P_{CH_3COOH})$$

The catalytic acetylene acetylation reaction is an exothermic process, $\Delta H^0_{298} = -98$ kJ/mol. The reaction is reversible. It was found that the activation energy of the synthesis of vinyl acetate from acetylene with the participation of the ZnO: CdO: ZrO_2/expanded clay catalyst is equal to E_a=29.2 kJ / mol. The reaction equilibrium constant and temperature have the following relationship:

$lgK_p = 4400/T – 7{,}22 \cdot lgT + 2{,}47 \cdot 10^{-3} \cdot T + 11{,}3$

where, T - temperature, K.

Table 4.Vinyl Acetate Yield and Equilibrium Constants

T, ^{O}C	T, K	lgK_p	K_p	X, %
150	423	3.783	6064.57	99.9
200	473	2.458	287	98.7

The influence of various factors on the reaction rate was studied in order to create an improved technology for the catalytic acetylation of acetylene into vinyl acetate. The influence of various factors on the reaction rate was studied in order to create acetylene kinetic acetylate, an advanced technology for the synthesis of vinyl acetate. It is proved that the yield of reagents increases with increasing concentration of acetylene in the

mixture, and with increasing partial pressure of acetic acid decreases. The introduction of vinyl acetate into the reaction mixture does not affect the rate of its formation.

With the introduction of water at temperatures above 210°C, hydrolysis of vinyl acetate increases, but does not affect the activity of the catalyst. With a molar ratio of acetylene: acetic acid of 4:1 and an increase in the process temperature of more than 200°C and a contact time of 4 seconds, the conversion of acetic acid almost reaches 100%. At a molar ratio of acetylene: acetic acid and temperature soaking at 200°C and reducing the contact time from 4 seconds to 2 seconds, the yield of vinyl acetate reaches 95-96%.

In order to calculate and study the optimal parameters of the process of studying the kinetics of the reaction in a differential reactor, a reactor model was created and the process was optimized, and an improved technology for catalytic acetylation and catalyst creation was proposed.

4. Optimization of the vinyl acetate synthesis process

In the synthesis of vinyl acetate from acetylene, the product yield from three factors: the process temperature (x_1), the space velocity of acetylene (x_2), and the height of the catalyst layer (x_3). A series of preliminary experiments was conducted on 2^3 full-factor experimental designs. The full coefficient is a matrix of the experimental design, its results are shown in Table 5.

Table 5. Complete Plan of the Factorial Experiment of the Plan Matrix and its Results

Transitional changes and degrees of factors		x_1	x_2	x_3	
Zero power $(x_i = 0)$		185	280	40	
Transitional change (Δx_i)		25	50	10	
Low degree $(x_i = -1)$		160	230	30	
High degree $(x_i = +1)$		210	330	50	
	Plan	x_1	x_2	x_3	Y
Experiences	1	-	-	-	20.5
	2	+	-	-	28.3
	3	-	+	-	26.8
	4	+	+	-	34.6
	5	-	-	+	26.4
	6	+	-	+	44.2
	7	-	+	+	52.7
	8	+	+	+	70.5

As a result of processing the experimental results, the following multifactorial results were obtained: $Y = 21 + 8x_1 - 16x_2 + 10x_1x_1$. To conduct analytical optimization of the model, we will have an equation of two linear types, if we consider the model as an integral equation and solve it optimally:

$$\partial y / \partial x_1 = 8 + 10x_2 = 0 \rightarrow x_2 = -\frac{8}{10} = -0{,}8$$

$$\partial y / \partial x_2 = 16 + 10x_1 = 0 \rightarrow x_2 = -16/10 = -1{,}6$$

In this case, the optimal equation takes the following form:

$$Y_{x_2=0,8} = 21 + 8x_1 - 16 \cdot 0,8 + 10x_1 \cdot 0,8 = 8,2 + 16x_1$$

The process of producing vinyl acetate from acetylene and acetic acid proceeds with the participation of $(ZnO)_x \cdot (CdO)_y \cdot (ZrO_2)_z$. The inclusion of ZrO_2 in the composition of the catalyst reduces the formation of additional reaction products and prevents their condensation due to the formation of resin in the porous catalyst, which leads to an increase in the operating time of the catalyst.

5. Conclusion

1. Based on the sol-gel technology for the catalytic acetylene acetylation reaction, a catalyst was created — a nanocatalyst with high catalytic activity $(ZnO)_x \cdot (CdO)_y \cdot (ZrO_2)_z$ / expanded clay.
2. The effect of various factors (temperature, space velocity, molar ratios C_2H_2: CH_3COOH, catalyst preparation method, etc.) on the yield of the target product in the acetylene acetylation reaction was studied.
3. Based on the results obtained, a kinetic model was compiled and the process of vinyl acetate synthesis was optimized based on this kinetic model.

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