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VINYLACETATE Production Out of ACETYLENE

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ABSTRACT: In the study, a nanocatalyst with high efficiency, activity and selectivity has been created for the catalytic acetylated reaction of acetylene on the basis of Sol-gel technology. The impact of various factors (temperature, volume velocity, C_2H_2:CH_3COOH mol ratio, the way of preparation of catalysts etc.) on vinyl acetate sulphide, selection process and conversion of starting material with the presence of a catalyst with the highest catalytic activity $(ZnO)_x{\cdot}(CdO)_y{\cdot}(ZrO_2)_z$/keramzite style has been studied. As a result of the research, the mechanism and kinetic equation of vinyl acetate steam phase is proposed. Based on the results obtained, the kinetic model has been created, on the basis of this kinetic model, the process of vinyl acetate synthesis has been optimized.

KEY WORDS: Acetic acid, Acetylene, Catalyst, Material balance, Optimization, Sol-gel technology, Vinyl acetate.

I.INTRODUCTION

The most important vinyl acetate is the vinyl acetate among oxygen-containing compounds that can be extracted from the major organic and petrochemical industries.

Vinyl acetate is widely used in industry as a monomer. One of the important properties of vinyl acetate is its polymerization. Polyvinyl acetate, polyvinyl alcohol and polyvinyl-acetal are extensively used among vinyl acetate polymer products produced from vinyl acetate. Polyvinyl acetate has high adhesion and elasticity properties, which is highly evaporative. Therefore it is broadly consumed in the manufacture of water-soluble latex paints, adhesives, fibers and others [1-2].

At the same time, one of the main tasks facing chemists in Uzbekistan consists of creating new, economical and wasteless methods and technologies of producing export-oriented and vital substances for national economy instead of imported ones, using local raw materials and waste gases and on this basis increasing the export potential of our country [3, 7-13].

The annual demand for vinyl acetate in the world is about 5 million tons while the annual demand for vinyl acetate in Uzbekistan is 30,000 tons. Currently, vinyl acetate is extracted from acetylene in Eastern and Western Europe and Asian countries. Evidently, one of the main directions of chemical production is the creation of selective, highly active catalysts in the development of waste or low-waste energy and resource-saving technologies.

II.RELATED WORK

The reaction of catalytic acetylation of acetylene was carried out in the flow reactor under the following optimal conditions: $T=180^0C$, $C_2H_2:CH_3COOH=4:1$, $V_{C2H2}=280$ hours^{-1} [4-6].

The reaction products were analyzed by means of chromatographic fluid in the following optimal conditions: Cvetochrom - 545, lithosphere with magnitude 0.250-0.315 nm, glass column with dimensions 2x0,004 m, columns temperature 100^{o}C, Carbon nitrogen flow rate is 30 ml / min. The quality analysis was carried out on the basis of comparisons of "eyewitnesses" and tidal size, quantitative analysis and internal regulation.

Information on the textural characteristics of the samples was obtained by low-temperature adsorption of liquid nitrogen at 77,35K in the ASAP 2010 M unit. Samples were dried for 4 hours at 120°C and burnt for 6 hours at 550°C.

The specific surface area was determined by the BET method. The total volume of umbilical cord was calculated on the maximum saturated adsorbed nitrogen content. The distribution by the size of the pelvis was determined by the method BJH (Barret-Joyner-Hallend).

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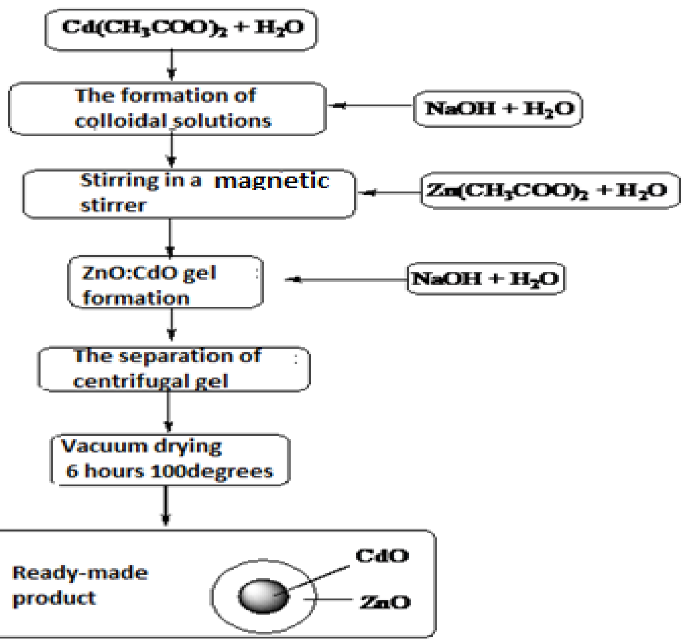
III.EXPERIMENTAL RESULTS AND THEIR DISCUSSIONS

Studies on the selection of catalyst were carried out under the standard conditions as follows: Reaction temperature 180^0C, volume acceleration over acetylene 280 hr^{-1}, acetylene : vinegar acid ratio = 4: 1.
The catalytic activity of catalysts prepared from salts of d-elements, prepared in "Sol-gel" technology for the first time in the catalytic acetylating reaction of acetylene in the steam phase was investigated (Table 1).

Table 1
Effect of initial substances on catalytic activity in acetylene catalytic acetylating reaction (T=180^0C, $C_2H_2:CH_3COOH$=4:1, V_{C2H2}=280 hr^{-1}, promotor: 1.8 % $K_2Cr_2O_7$)

№	Catalyst Structure	Conversion of CH_3COOH, %		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

As shown in Table 1, the catalyst (№4) containing zinc, cadmium and zirconium oxides has high selectivity and efficiency.
As can be seen in Table 1, the total conversion of acetic acid to 85.4%, conversion to vinyl acetate - 79.8% under the selected optimal reaction conditions with the participation of catalyst №4.
Nucleus/shell nanocatalyst was selected for acetylene and vinyl acetate acetic acid. The choice of nanocatalyst is carried out according to the following scheme.
Core/shell structured ZnO:CdO nanoscale synthesis scheme:



Picture 1. Nucleus/Shell Structured ZnO: CdO nanoscale synthesis scheme

The total volume of catalyst pores is 0.3-0.41 cm^3/ g, specific surface area is 50-170 m^2/ g.
The ceramic catalyst was used as a catalyst holder, its surface was 690-720 m^2/g, the total volume of pores - 0.87-0.92 cm^3/g, the size of the microwave - 0.24-0.25 cm^3/g. Granules have a diameter of 200-500 mcm. The acetylene

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acetylating reaction was carried out at 170-220^{0}C, the volume of acetylene was 0.54-0.84 l/cm^3(h), and the mass consumption of acetic acid was 0.3-0.5 g/cm^3 (h).
The catalyst flow cuvette with a diameter of 9 cm3 was dropped to the reactor and the system was washed at nitrogen flow at 15 l/h for 10 minutes. Vinyl acetate synthesis was held at atmospheric pressure at 180^{0}C. In the above conditions, the catalyst's working limit was 2000 hours.
Then we learned the effect of mass concentrations of the active ingredient №4. The results are shown in Table 2.

Table 2

Effect of mass relativity of catalyst active components on catalytic activity in acetylene

№	Catalyst composition				Catalyst productivity, g/l hour
	$Zn(CH_3COO)_2$: $Cd(CH_3COO)_2$	$Zn(CH_3COO)_2$, mass, %	$Cd(CH_3COO)_2$, mass, %	$ZrO(NO_3)_2$ mass, %	
1	9:1	25.6	2.8	-	205
2	9:1	23.7	2.6	1.0	328
3	9:1	21.8	2.4	2.0	316
4	3:1	20.0	6.7	-	230
5	3:1	18.6	6.6	1.0	363
6	3:1	17.5	5.8	2.0	347
7	1:1	15.0	15.0	-	315
8	1:1	13.5	13.5	0:1	440
9	1:1	11.5	11.5	1.0	450
10	1:1	11.0	11.0	2.0	430
11	1:1	10.0	10.0	5.0	418

As shown in Table 2, the catalyst productivity is highest when the mass ratio of the active components of the catalyst is Zn $(CH_3COO)_2$: Cd $(CH_3COO)_2$: $ZrO(NO_3)_2$ = 11.5: 11.5: 1.0 (mass percentages).
The effects of various factors (temperature, volume velocity, C_2H_2:CH_3COOH mole ratio, catalyst preparation method) for vinyl acetate productivity, selection process and conversion of the starting material with the aid of the catalyst №4 shown in Table 1 are examined.
When studying the effect of C_2H_2:CH_3COOH on vinyl acetate yield and process selectivity, the most favorable conditions were C_2H_2:CH_3COOH ratio of 4: 1 (Table 3).

Table 3

Effect of C_2H_2:CH_3COOH ratio on Vinyl acetate Flue

(T=180^{0}C, catalyst № 4)

№№	C_2H_2:CH_3COOH mole ratio	Conversion of acetic acid,%		Selectivity, S %
		General	Vinyl acetate	
1	1:3	48.0	18.4	38.3
2	1:2	63.4	48.5	76.5
3	1:1	78.8	63.2	80.2
4	2:1	82.0	70.7	86.2
5	3:1	83.8	75.4	90.0
6	4:1	85.4	79.8	93.4
7	5:1	92.5	72.0	77.8
8	6:1	96.2	65.4	68.0

As can be seen from the table, the total conversion of acetic acid rises with the increase in the mole amount of acetylene in the reaction mixture. The ratio of vinyl acetate to yield is reduced by the addition of the additives (etylidene acetate) when the ratio of the starting material to the ratio of 4: 1 is greater.
Temperature effect of acetic acid vinyl acetate reaction in the presence of catalyst №4 was studied in the range of 150-240°C (Table 4). When the temperature of the vinegar reaction to vinyl acids in the range of 150-240^{0}C was increased, vinyl acetate was increased to 185^{0}C, and the reaction rate decreased due to the decomposition of vinyl acetate and the addition of the additives when the temperature 185^{0}Cexceeded .

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As you can see from Table 4, as the temperature rises, the total conversion of acetic acid increases. Vinyl acetate is increased to 180°C and the temperature decreases when it exceeds 180°C. When the temperature exceeds 180°C, the reaction rate decreases due to the disintegration of the vinyl acetate and the addition of additives.

As a result of the research, in the case of the conversion of vinyl acetate to 0.005% crotone aldehyde, the reaction slows the polymerization of vinyl acetate by 15% and by 0.2% to 60% by the crotone aldehyde.

Table 4

Effect of temperature on acetylene acetylating reaction

(C_2H_2:CH_3COOH=4:1, $V_{C_2H_2}$ =280 hr^{-1} № 4)

№	Temperature, ^0C	Conversion of CH_3COOH,%		Selectivity S %
		General	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

With the help of the selected catalysts, the effect of particulate pressure on the reaction rate was investigated in a wide range of variables to investigate the mechanism and kinetics of the catalytic reaction of acetylene. The experiments were carried out at a constant value of the reaction rate of the reaction gas, which was due to the addition of the inert gas argon. As a result of the research, a decrease in vinyl acetate content was detected by an increase in the amount of acetic acid and a reduction in the partial pressure of the acetylene.

Based on the results of experiments and chromatographic analysis and literature data, the following mechanism of reaction was suggested:

$$I. CH_3COOH + Z \rightarrow CH_3COOH * Z$$
$$II. CH \equiv CH + Z \rightarrow CH \equiv CH * Z$$
$$III. CH_3COOH * Z \rightarrow CH_3COO^- * Z^+H^+$$
$$IV. CH \equiv CH * Z + H^+ \rightarrow CH_2 = CH^+ * Z$$
$$V. CH_2 = CH^+ * Z + CH_3COOH \rightarrow CH_2 = CHOCOCH_3 * Z + H^+$$
$$VI. CH_2 = CHOCOCH_3 * Z \rightarrow CH_2 = CHOCOCH_3 + Z$$
$$------------------- vinylacetate$$
$$VII. CH_2 = CH^+ * Z + CH_3COOH * Z \rightarrow CH_2 = CHOH * Z + CH_3C^-O * Z$$
$$VIII. CH_2 = CHOH * Z \rightarrow CH_3CHO + Z$$
$$--------------- acetaldehyde$$
$$IX. CH_3C^-O * Z + CH_3CHO \rightarrow CH_3OCOCH(CH_3)O^- * Z$$
$$X. CH_3OCOCH(CH_3)O^- * Z + CH_3C^+O * Z \rightarrow CH_3CH(OCOCH_3) + 2Z$$
$$--------------------------- etylidendiacetate$$
$$XI. CH_3C^+O * Z + CH_3CHO \rightarrow CH_3OCOC^+H(CH_3)_2 * Z$$
$$XII. CH_3OCOC^+H(CH_3)_2 * Z + CH_3COO^- \rightarrow CH_3CH(OCOCH_3)_2 * Z$$
$$XIII. CH_3CH(OCOCH_3)_2 * Z \rightarrow CH_3CH(OCOCH_3)_2 + Z$$
$$------------------ etylidendiacetate$$
$$XIV. CH_3COOH * Z + CH_3COOH * Z \rightarrow 2Z + CH_3COCH_3 + CO_2 + H_2O$$
$$---------------------------- acetone$$
$$XV. CH_3COO^- * Z + CH_3COO^- * Z \rightarrow CH_3OCOCOCH_3 + 2Z$$
$$---------------------- acid\_anhydride$$

A) Active centre of Z-catalyst.

The proposed mechanism confirms and supplements the known theories from scientific literature.

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To interpret the obtained experimental data, it is necessary to find kinetic equations that satisfy the variation of parameters (reaction speed, reaction velocity, adsorbent coefficient, and partial pressure) in a wide range. After identifying the kinetic parameters of these equations, one can think of which of them satisfies the experimental data.
It is known from scientific literature that the kinetics of the catalytic acetylating reaction of acetylene are studied in the following conditions and in 3 types of catalysts. Considering the above, the following kinetic equations were chosen for acetylene acetylating reaction and corresponding parameters were calculated:

1. $Zn(OAc)_2/C$; ($t=180\text{-}220^0C$); $W=K P_{C_2H_2} /(1+b P_{CH_3COOH})$
2. $ZnO/\gamma Al_2O_3$; ($t=230\text{-}270^0C$); $W=K P_{C_2H_2} \cdot P_{CH_3COOH}$
3. $Cd(OAc)_2/\gamma Al_2O_3$; ($t=170\text{-}230^0C$); $W=K P_{C_2H_2} \cdot P_{CH_3COOH} /(1+b P_{CH_3COOH})$

The adequacy of the above equations was investigated to determine which kinetic equation satisfies the reaction. Depending on the given equation and experimental results, the kinetic equation parameters were determined by minimizing the theoretical calculated value from the sum of squares of experimental values. As a criterion for the adequacy of the kinetic equation, the following condition is fulfilled:

$$\sum_{i=1}^{n} (W_{practical} - W_{theoretical})^2 \Rightarrow \min .$$

The adequacy of the equations is based on the average square value (S) of the difference between the obtained and theoretical calculations. The determined kinetic constants and adsorption coefficients were used to calculate the velocity of vinyl acetate synthesis reaction from acetylene using different kinetic equations. The acetylene acetylating reaction satisfies the following equation according to the results of chromatographic analysis and the study of the effects of particulate pressure, volume velocity and temperature on the velocity of acetylene acetylating reaction:

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

The acetylene catalytic acetylene reaction is an exothermic process, $\Delta H^0_{298} = -98$ kJ/mol. Reaction regenerate. The active energy of the process of acetylene synthesis from vinyl acetate with $ZnO:CdO:ZrO_2$/ceramic catalyst was determined to be $E_a = 29.2$ kJ/mol.
The balanced constant of the reaction has the following relationship with temperature:

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

there is T-temperature, K.

Table 5
Constant of vinyl acetate productivity and balance

T, ^0C	T, K	$\lg K_p$	K_p	X, %
150	423	3.783	6064.57	99.9
200	473	2.458	287	98.7

Modeling vinyl acetate synthesis reactor. Most process technology is fully dependent on the activity of catalysts. The activity and selectivity of the catalysts ensure the rate and quality of catalytic reaction. Synthesis of acetylene and acetic acid from vinyl acetate was carried out in an ideal tissue reactor with the presence of an inert layer lubricant. Considering the deactivation of the catalyst, the mathematical model of the reactor for acetylene and vinyl acetate synthesis can be described as follows:

$$\frac{\partial x}{\partial l} = \frac{\Theta(v+1)}{v} r(m, V_{volume}, T, x);$$

$$\frac{\partial \Theta}{\partial t} = \Theta k_p \quad (T)$$

In the conditions of approach:

$$x(0,t) = 0; \quad t \in [0, t_k]; \qquad \Theta(\ell,0) = \Theta_0; \quad \ell \in [0, \ell_p]$$

There is V - the volume ratios of acetylene and acetic acid; v_{volume} - Volume velocity of raw materials consumption; t - catalyst's effect time; Θ - the magnitude of the catalytic activity change; T - temperature ($T \le 180^0 C$); ℓ_p -reactor length; t_k - catalyst's working time.

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As a result, the basic parameters of the tubular reactor were determined in the production of vinyl acetate (Table 6).

Table 6
Main parameters of working reactor in vinyl acetate production

$T, ^0C$	P, MPa	$U_{g\cdot p\cdot mg}$, m/s	Heat carriage	G, 1000 tons/year	D, м	h,m	N
170-200	0.1	0.60	Boiled water	50	2.5	6.5	4.7

B) Material balance of vinyl acetate production. Synthesis of vinyl acetate from acetylene and acetic acid leads to the following reaction:

$$C_2H_2 + CH_3COOH \leftrightarrow CH_3COOCH = CH_2;\ \Delta H^0_{298} = -98\ kJ/mol$$

As catalysts are used zinc acetate, cadmium acetate, or mixtures thereof, which are separated into different tanks (activated charcoal, silica gel, aluminum oxide, pumice, etc.). The catalyst used is satisfactory at 180-270^{0}C.
The catalyst is deactivated by zinc acetate displacement and zinc acetate as a result of the following reaction:

$$(CH_3COO)_2Zn \rightarrow ZnO + CO_2 + CH_3COCH_3$$

The resulting zircon oxide is inactive in the main reaction. However, under reaction conditions, acetic acid reacts with zinc acetate:

$$ZnO + 2CH_3COOH \rightarrow Zn(CH_3COO)_2 + H_2O$$

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

$$2CH_3COOH \rightarrow (CH_3)_2CO + H_2O + CO_2$$
$$C_2H_2 + H_2O \rightarrow CH_3CHO$$
$$2C_2H_2 + H_2O \rightarrow H_3C\text{-}CH\text{=}CH\text{-}CHO$$
$$CH_3COOH + CH_3COOCH\text{=}CH_2 \rightarrow CH_3CH(OOCCH_3)_2$$
$$CH_3COOCH = CH_2 + H_2O \leftrightarrow CH_3COOH + CH_3CHO$$

In order to avoid additional reactions, the reaction is carried out with a high concentration of acetylene.
Acetylene and producing vinyl acetate from acetic acid can be described by the following general equation:

$$CH\equiv CH + CH_3COOOH \leftrightarrow CH_3COOOCH\text{=}CH_2 \quad (1)$$

The reaction takes place at 180^{0}C and normal atmospheric pressure.

Table 7
Material balance of vinyl acetate production

$C_2H_2 \div CH_3COOH$=4:1; $V_{C_2H_2} = 280\ hour^{-1}$;

$K_{CH_3COOH} = 85.4\%$; $K_{CH_3COOH} \rightarrow BA(79.8)$; **cat :$(ZnO)_x\cdot(CdO)_y\cdot(ZrO_2)_z$**

Components	Before reactor		After reactor	
	kg/hr	k mole/hr	kg/hr	k mole/hr
Technical acetylene, from it:	338.542			
Pure acetylene	325	12.5	241.54	9.29
Additives	13.542		13.542	
Frozen acetic acid (96%) from it:	195.3125			
Pure acetic acid	187.5	3.125	27.375	0.456
H_2O	7.8125	0.434	-	-
Vinyl acetate	-	-	183.15	2.13
Acetone	-	-	13.867	0.239
Acid aldehyde	-	-	20.311	0.462
Crotone aldehyde	-	-	10.963	0.157
Etylidendiacetate	-	-	14.6	0.1
CO_2	-	-	8.5	0.193
Total	533.850	16.06	533.848	13.03

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IV.CONCLUSION

1. A catalytic nanocatalyst with a high catalytic activity $(ZnO)_x{\cdot}(CdO)_y{\cdot}(ZrO_2)_z$ / keramzite composition was created for the catalytic acetyl acetate reaction of acetylene on the basis of "Sol-gel" technology.
2. The catalytic reaction of acetylene acetylene yield of the target product of various factors (temperature, speed, volume C_2H_2:CH_3COOH ratios, such as the method of preparation of the catalyst) studied the effect.
3. On the basis of the obtained results a kinetic model was created, based on this kinetic model, the process of vinyl acetate synthesis was optimized.
4. Selected high catalytic activity $(ZnO)_x{\cdot}(CdO)_y{\cdot}(ZrO_2)_z$/ keramzite components of the textile characteristics of the catalyst and the process of synthesis of vinyl acetate studied the kinetic laws. As a result of the research, the mechanism and kinetic equation of vinyl acetate steam phase was proposed.

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