



# PHYSICO-CHEMICAL CHARACTERISTICS OF VINILATECETATE SYNTHESIS CATALYSTON FROM ACETYLENE

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## ABSTRACT

The catalyst was prepared under the following conditions: 5-25% solutions of zinc acetate and cadmium acetate and 1-3% solution of zirconyl nitrate were injected into the microspheric nanoparticle carrier (expanded clay) by circulating absorption at 60°C. The absorption time of the salts was varied between 60-90 minutes. Subsequent analysis of the IR spectrum showed that as the heating temperature of the sample increases, there will be changes in the IR spectra in the area of valence oscillations (2800-3900 cm<sup>-1</sup>) and in the area of deformation oscillations (4000-900 cm<sup>-1</sup>).

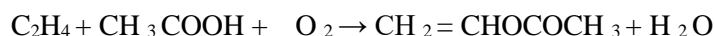
**KEYWORDS:** acetylene, acetic acid, vinyl acetate, catalyst, sol-gel technology, material balance, flow chart.

## INTRODUCTION

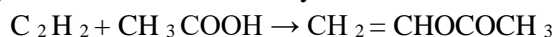
Polyvinyl acetate is a polymer with high adhesive properties, which is widely used in the production of adhesives, water-soluble latex dyes and fabric reinforcement. However, polyvinyl acetate, polyvinyl alcohol is important in medicine, agriculture, in the creation of synthetic rubbers, synthetic fibers, biologically active substances, modified polymer compounds and other unique properties materials [1-4].

Currently, the production of vinyl acetate in developed countries is carried out in two ways:

1. Oxidation-based esterification of ethylene (Moiseev reaction). The process is based on the reaction between ethylene, acetic acid and oxygen in the presence of a catalyst [5]:



2. Based on the vapor phase catalytic reaction between acetylene and acetic acid [6-12]:



The first method is common because the cost of ethylene is cheaper than acetylene. Inexpensive acetylene sources are currently being found as an adjunct to new manufacturing processes. Therefore, the production of vinyl acetate from acetylene remains promising.

Acetylene, ethylene or acetaldehyde is used as a raw material for the production of vinyl acetate. The competitiveness of a particular method is largely determined by the availability and cost of the starting reagents. The bimetallic Pd-Au catalyst system is used in the ethylene method and has good performance in industrial production [13]. Miyazawa et al synthesized several highly active metal oxide catalysts, but there are clear shortcomings in terms of stability.

Today, the annual demand for vinyl acetate in the world is 8 million tons. This requires research to create a system for optimizing the technology of obtaining vinyl acetate and its derivatives with the participation of local



catalysts. Therefore, it is important to conduct research on the development and improvement of technology for the production of vinyl acetate with the participation of local catalysts.

### EXPERIMENTAL PART

For the production of vinyl acetate from acetylene and acetic acid, solutions of zinc acetate, cadmium acetate and zirconium nitrate were ingested into a microspheric nano-porous expanded clay with a size of 200-500  $\mu\text{m}$ . The total volume of pores of the catalyst is 0.3-0.41  $\text{cm}^3/\text{g}$ , the specific surface area is 50-170  $\text{m}^2/\text{g}$ .

The catalyst was prepared under the following conditions: 5-25% solutions of zinc acetate and cadmium acetates and 1-3% solutions of zirconyl nitrate were absorbed into the microspheres nanoporous carrier (expanded clay) by circulating absorption at 60 ° C. The absorption time of the salts was varied between 60-90 minutes. Absorption carrier (expanded clay): solution was carried out in a ratio of 1: 3-1: 8.5. At the end of the absorption process, the catalyst was dried at room temperature for 24 h and then in a drying oven at 100–130 ° C (raising the temperature to 10 ° C every 1 h). The content of zinc acetate in the catalyst was 11-30% [86].

### EXPERIMENTAL RESULTS AND THEIR DISCUSSION

The texture characteristics of the nanocatalyst based on Zol-gel technology are given in Table 1 [95, P. 4923-4930 .].

**Table 1.  $(\text{ZnO})_x(\text{CdO})_y(\text{ZrO}_2)_z$ /texture characteristics of expanded clay catalyst**

No	Granular shape	Density, g / $\text{cm}^3$	$S_{\text{col}}$ $\text{m}^2/\text{g}$	The total volume of the pore, $\text{cm}^3/\text{g}$	Amounts of ZnO: CdO: ZrO <sub>2</sub> , in% of mass
1	ball	0.98	51	0.310	10.3: 2.6: 0.4
2	ball	0.94	63	0.341	9.6: 2.2: 0.5
3	ball	0.88	46	0.253	12.0: 2.6: 0.3
4	ball	0.76	107	0.362	11.3: 2.1: 0.6
5	ball	0.72	173	0.409	11.8: 2.5: 0.2
6	ball	0.86	57	0.337	9.2: 2.0: 0.1
7	ball	0.79	62	0.313	9.0: 2.5: 0.5
8	ball	0.87	59	0.329	8.5: 1.5: 0.2
9	ball	0.88	70	0.318	10.8: 3.0: 0.4
10	ball	0.92	51	0.310	8.8: 2.2: 0.25
11	ball	0.85	59	0.271	9.5: 1.5: 0.3
12	ball	0.85	51	0.240	10.2: 1.86: 0.4
13	ball	0.91	58	0.269	11.5: 2.4: 0.6
14	ball	0.95	68	0.320	16.7: 2.1: 0.3

Investigations obtained by the adsorption-desorption method of N<sub>2</sub> show that ZnO powder heated at 500 ° C is meso-porous with a pore size of 7-9 nm (shown in Figures 1-2) [97; P. 1691–1701.].

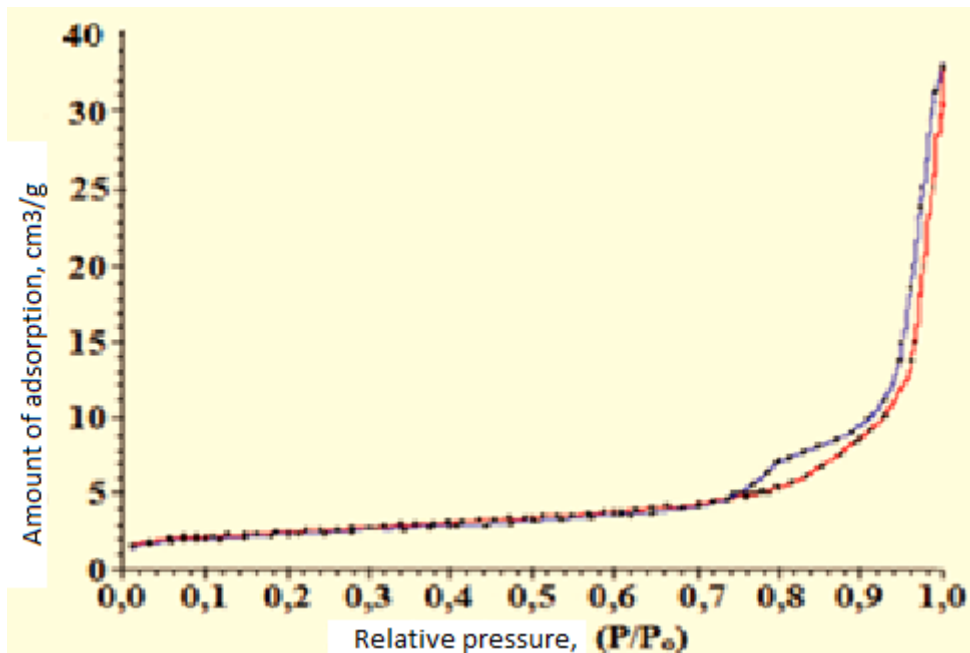


Figure 1. Adsorption-desorption of N<sub>2</sub> in ZnO

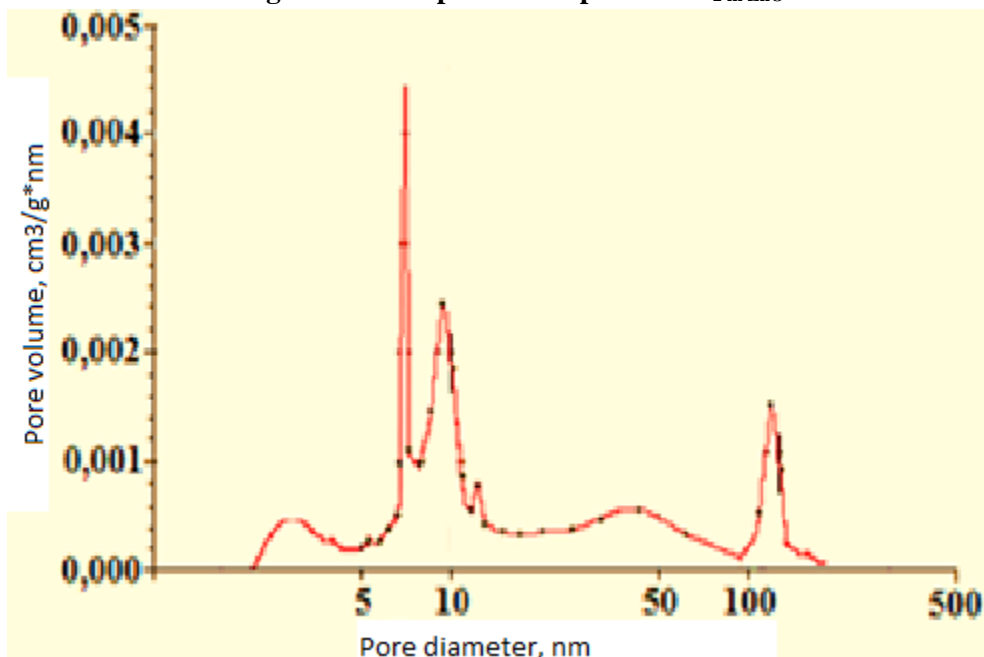
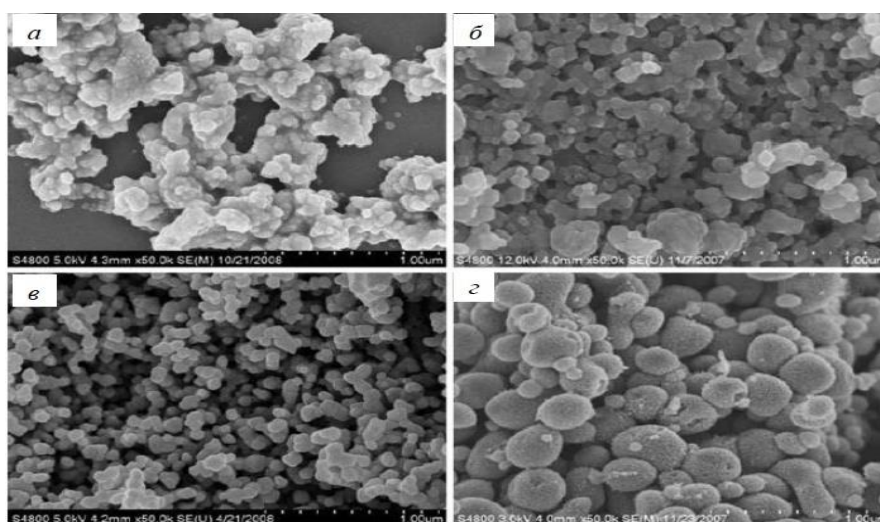


Figure 2. Distribution of pores by size for ZnO heated at 500 °C.

When heated to 700 °C, the specific surface area decreases from 8.6 to 3.3 m<sup>2</sup>/g due to the interconnection (spekanie) of the particles (Figures 4). As a result, a new porous structure is formed and the size of the mesopores is 10-50 nm. Macropores are also formed.

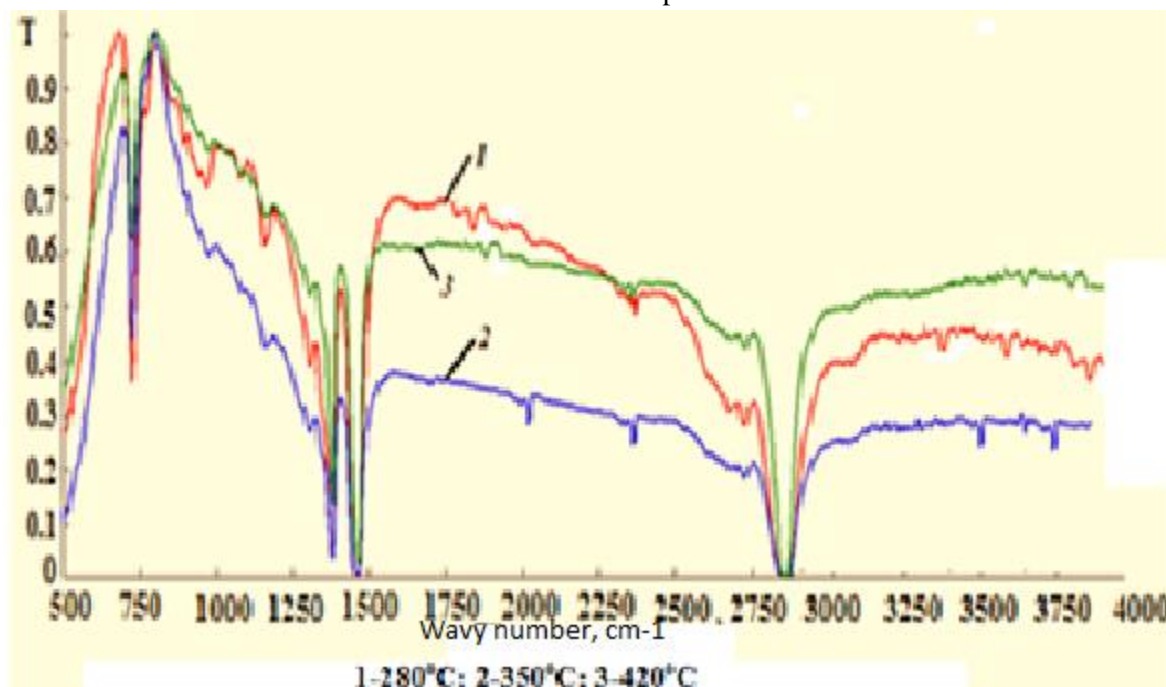
Analysis of the IR spectrum of a sample of zinc oxide dried at 125 °C and fired at 350, 450, 550 °C shows that the sample contains 1020-1067 cm<sup>-1</sup> hydroxyl corresponding to the absorption area and 725, 1332, 1400, 1550, 2880- It was found that there was an acetate group corresponding to the absorption area of 2920 cm<sup>-1</sup>. Also chemically and physically adsorbed water (677, 877, 918, 1550, 3145-3425 cm<sup>-1</sup>), amino group (677, 877, 1550 cm<sup>-1</sup>) and Zn (H<sub>2</sub>O)<sub>2</sub><sup>2+</sup> and Zn (NH<sub>3</sub>)<sub>2</sub><sup>2+</sup> groups are also available.



**Figure 4. Microphotography of ZnO obtained under a light-emitting electron microscope using a structure-forming agent (a), ethylenediamine (b), hexamethylenediamine (c), and citric acid.**

Subsequent analysis of the IR spectrum showed that with increasing heating temperature of the sample, there will be changes in the IR spectra in the area of valence oscillations ( $2800-3900\text{ cm}^{-1}$ ) and in the area of deformation oscillations ( $4000-900\text{ cm}^{-1}$ ).

In the IR spectra of ZnO samples of nanoscale heated at  $400-750\text{ }^{\circ}\text{C}$ , a clearly expressed maximum is observed at  $450\text{ cm}^{-1}$ . This is related to the Zn-O bond oscillation in the spectrum.



**Figure 5. Of nanostructured ZnO at different temperatures**

We then examined the IR spectrum by burning the sample from  $125\text{ }^{\circ}\text{C}$  to  $750\text{ }^{\circ}\text{C}$ . Analysis of the IR spectrum of a sample of zinc oxide dried at  $125\text{ }^{\circ}\text{C}$  shows that the sample contains a hydroxyl corresponding to the absorption area of  $1020-1067\text{ cm}^{-1}$  and an acetate group corresponding to the absorption area of  $725, 1332, 1400, 1550, 2880-2970\text{ cm}^{-1}$  became known. Also chemically and physically adsorbed water ( $677, 877, 918, 1550, 3145-3425\text{ cm}^{-1}$ ), amino group ( $677, 877, 1550\text{ cm}^{-1}$ ) and  $\text{Zn}(\text{H}_2\text{O})_2^{2+}$  and  $\text{Zn}(\text{NH}_3)_2^{2+}$  groups are also available.

Figure 6 shows diffractograms of ZnO / CdO nanocomposites. As can be seen from the figure, the presence of corresponding diffraction peaks corresponding to ZnO and CdO nanocomposites is indicated. For each ZnO / CdO nanocomposite calcination temperature, the peaks and positions of the constitutional phases were obtained, which were adjusted using JCPDS reference data [CdO 05-0640 and ZnO 79-2205]. The reflection peaks of ZnO appear as index

planes (100), (002), (101), (102), (110), (103) and (201), 31.7, 34.3, 36.2, 47, 4, 56.5, 62.7 and 69.0. The diffraction peaks of CdO are indexed with values (110), (200), (220), (311) and (222) at the corresponding 2 $\theta$  values of 33.0, 38.3, 55.3, 65.9 and 69.3, respectively. ZnO / CdO, as well as ZnO / CdO nanocomposite samples heated to different temperatures, have a mixed hexagonal structure of the ZnO phase and a cubic structure of the CdO phase. These structures are compatible with standard card diffraction of ZnO and CdO nanoparticles. In this study, the formation and formation of the crystal structure of the ZnO / CdO composition is consistent with previous literature.

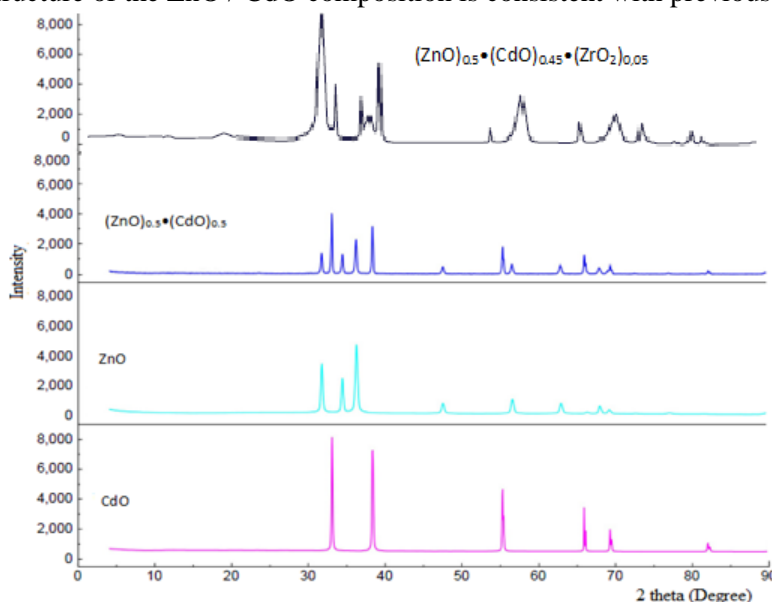


Figure 6. CdO, ZnO, (ZnO)<sub>0.5</sub> • (CdO)<sub>0.5</sub> and (ZnO)<sub>0.5</sub> • (CdO)<sub>0.45</sub> • (ZrO<sub>2</sub>)<sub>0.05</sub> radiographs of samples

## CONCLUSION

The method of preparation of catalysts on the technology "Zol-gel" and the texture characteristics of the obtained catalyst were studied. Studies have shown that the synthesized nanocrystals are composed of agglomerates, the average size of which increases with increasing firing temperature.

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