

CHROMATOGRAPHIC ANALYSIS OF PRODUCTS OF THE STEAM-CARBONATE CONVERSION REACTION OF METHANE

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Abstract

The catalytic carbonation reaction of methane with carbonic anhydride in the gas phase in the presence of catalysts was carried out in a flow reactor (diameter 2 cm and length 20 cm), at normal atmospheric pressure, a volume flow rate of 1000 h-1 and at 750-1000°C. The catalyst volume was 6 ml, and the catalyst operating time was 240-264 h.

Introduction

The gas and liquid products of the catalytic carbonation reaction of methane with carbon dioxide in the gas phase in the presence of catalysts were qualitatively and quantitatively analyzed by gas chromatography. All work on gas chromatography determination was carried out on chromatographs "Gazokhrom 3101" and "Tsvet 100, model 165" [72].

During the catalytic carbonation of methane with carbon dioxide in the gas phase in the presence of catalysts, a mixture of gases consisting of nitrogen, hydrogen, oxygen, carbon oxides, and methane is formed. Artificial mixtures of gases were prepared to select the optimal conditions for the separation of this mixture. Activated carbon and polysorb were used as sorbents to separate them in a chromatographic column. The factors influencing the selection of optimal conditions for the separation process were the column size (from 1 m to 3 m), the mobile phase flow rate (from 20 to 50 ml/min), and the size of the sorbent particles. -(0,150-0,500 mm), column thermostat temperature -(room temperature) 120°C up to), and as a criterion, the size-separation level (RS) indicating the degree of mutual separation of the mixture of substances (components) was taken. Based on the results of the experiment, a regression equation of the following form was created, which represents the dependence of the separation rate (RS) on the factors influencing the separation: and assessed the adequacy of Eq. The optimal values of the factors that ensure high separation of the mixture of substances (components) were carried out by the simplex method of optimization based on the adequate regression equation.

Deactivation of catalysts and their regeneration

The main disadvantage of the created catalyst is that they are coked (active centers with coke) and condensation of reaction by-products leads to the formation of resinous substances, which clog the catalyst pores, that is, they lose their catalytic activity and become inactive. Therefore, catalyst regeneration is required.



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If the C/H ratio is between 1.5 and 2, the coke produced is graphitized in structure and its heating is carried out at a sufficiently high temperature (around 800-900°C). If the C/H ratio is between 0.2 and 1, amorphous coke is produced..

The combustion of graphite, the main combustible component of coke, is characterized by the following processes:[74. 12-21-p. 91-106-p.]

1) Carbon can be partially or completely oxidized when it reacts with oxygen:

 $C+O_2 \rightarrow CO_2+395,4 \text{ кж/мол;}$

$$C+1/2O_2 \rightarrow CO+110,4$$
 кж/мол

3) Graphite can react with water vapor in the reaction zone:C+H₂O→CO+H₂+41,0 кж/мол In general, the equation for the combustion reaction of coke is:

$$CH_x + \left(1 + \frac{x}{4}\right)O_2 \rightarrow CO_2 + \frac{x}{2}H_2O$$

The amount of carbon dioxide produced by the combustion reaction of the coke produced by the carbonation reaction of this methane in the gas phase w asdetermined as follows:

$$n_{O_2} = \frac{G_{xx6x} (C_{O_2}^0 - C_{O_2})}{100 V_m} \qquad ea \qquad n_{CO_2} = \frac{G_{xa60} (C_{CO_2} - C_{CO_2}^0)}{100 V_m}$$

The results obtained on the physicochemical properties of the catalysts are presented in Table 1 below.

Table 1

Catalyst	Relative surface area,	Average	pore	Pore size, cm $^3/\Gamma$
	M^2/Γ	diameter, nm		
	47÷75	11÷13нм		0,1262÷0,1803

The amount of coke in the catalysts was determined in two ways: by the difference between their masses before and after heating in open air. For this, the catalyst was heated in air at a temperature of 110°C and its mass was measured, then placed in a furnace at 800°C. After 1 hour, its mass was measured again and the amount of coke was determined. The amount of carbon in the catalyst created by the second method was determined by quantitatively oxidizing it to SO2. The following formula was used to calculate the amount of coke (in mass %) in the catalyst: $C = \frac{K \cdot S_c \cdot 100}{P}$

where K is the calibration factor determined by the standard substance, MT/MM²; S_c -The surface area of the CO2 peak obtained when the substance under study is burned; P- mass of the substance sample under research method, μg . Accuracy of the method ± 0.1 mass is %. [75;12-22-6].



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Summary

The amount of carbon dioxide produced by the combustion reaction of the coke produced by the carbonation reaction of this methane in the gas phase w as determined as follows: The amount of coke in the catalysts was determined in two ways: by the difference between their masses before and after heating in open air. For this, the catalyst was heated in air at a temperature of 110°C and its mass was measured, then placed in a furnace at 800°C. After 1 hour, its mass was measured again and the amount of coke was determined...

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