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MECHANISM AND KINETICS OF THE OXIDATION PROCESS OF NH_3, H_2S AND CH_4 IN THE PRESENCE OF SEMICONDUCTOR GAS SENSITIVE NANOCOMPOSITE METAL OXIDES

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Abstract. In the work, the laws of oxidation with metal oxides of hydrogen sulfide, ammonia, and methane were studied. The composition of the reaction products with the presence of highly active catalytic systems prepared on the basis of iron, titanium, copper, tungsten, and zinc oxides was investigated, and the mechanism of the oxidation process and optimal temperature ranges were proposed. On the basis of the obtained materials, gas-inert materials for competitive, selective semiconductor sensors of ammonia, hydrogen sulfide and methane were created and their performance was determined.

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Introduction. Today, ammonia, hydrogen sulfide, and CH_4 are among the most common toxic and explosive components released into the atmosphere in industrial waste gases [1-5]. Chemical sensors are widely used in the control of toxic and explosive compounds in various sectors of the economy [6,9]. Scientific research aimed at developing semiconductor materials and highly selective gas sensors is being conducted in the world's leading scientific centers [6-8] .

In particular, at Samarkand State University, research is being conducted on the development of sensitive and selective semiconductor sensors for various gases and the selection of the optimal composition of gas-insensitive materials (GSM) for these sensors [9-11]. In this regard, it is of particular importance to determine the laws and optimal conditions for the formation of selective GSMs based on sol-gel technology using metal oxides[12-15] and to develop high-efficiency semiconductor sensors (SCS) and study their metrological description[16-19]. earns.

The aim of current study of process laws of formation of gas-sensitive materials for chemical sensors using sol-gel technology. Based on the obtained materials, it consists in determining the kinetics and mechanism of the oxidation process of ammonia, hydrogen sulfide and methane.

Experimental.

The schematic diagram of the apparatus for studying the laws of ammonia, hydrogen sulfide, and methane oxidation processes used in the research is shown in Fig. 1.1. The apparatus consists of a cylinder filled with a

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gas mixture (1), a quartz tube (2), a layer of catalyst (3), a gas dispenser (4), and a gas chromatograph (5). and consists of an absorbing glass (6).

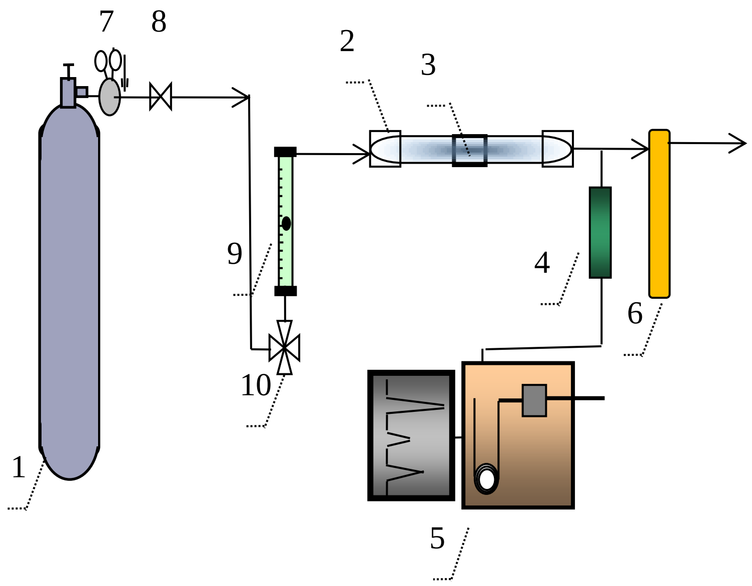

Figure 1.1. A device for studying oxidation laws of gases in the presence of semiconducting metal oxides. Gas mixture filled cylinder (1), quartz tube (2), catalyst (3), gas dispenser (4), gas chromatograph (5) and absorbing glass (6), reducer (7), gas throttle (8 and 10), ratiometer(9).

Modern gas chromatographic, photocolorimetric and potentiometric methods were used to determine the composition of initial products and products resulting from the reaction.

Results and discussion.

Taking into account the results of the study of the laws of oxidation in the presence of various metal oxides and the resistance of oxides to the effects of detectable components, the following semiconductor oxides were selected for the gas-sensitive material of the semiconductor sensor of ammonia, hydrogen sulfide and methane:

For NH_3 - TiO_2, Cr_2O_3 (Fe_2O_3);

For H_2S - WO_3, CuO;

For CH_4 - ZnO, CoO.

The conditions of oxidation of ammonia, hydrogen sulfide and methane in the presence of metal oxides in different proportions were studied. In these experiments, the results observed in the presence of Cr_2O_3:TiO_2 are numerically close to the results observed in the presence of Fe_2O_3:TiO_2 and have the following values: in the ratio from 10:90 to 90:10, the oxidation of ammonia is -40%, of which in the range from 10:90 to 50:50 34 % and in the range from 50:50 to 90:10 a reduction of NH_3 oxidation by -5% was observed. Thus, as a result of studying the activity and selectivity of individual and binary metal oxides during the oxidation of combustible gases, the composition of composites consisting of n- and p-type semiconducting metal oxides was selected for the gas-sensitive material of semiconductor sensors of NH_3, H_2S and CH_4. Also, as a result of the conducted research, a ratio of 10:90 of binary

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metal oxides with high activity and selectivity in oxidizing NH_3, H_2S and CH_4 was determined. In the presence of these nanocomposite mixtures, high activity and selectivity of the oxidation process of NH_3, H_2S and CH_4 are ensured. In further studies, the kinetics and mechanism of the oxidation process of NH_3, H_2S and CH_4 were studied.

a). Mechanism and kinetics of oxidation of NH_3 in the presence of 90%TiO_2+10%Fe_2O_3. Catalytic oxidation of ammonia is one of the important processes in heterogeneous catalysis. Because this process can go in the following three directions [20]:

$$2NH_3+1(1/2)O_2=N_2+3H_2O \quad (1)$$

$$2NH_3+2O_2=N_2O+3H_2O \quad (2)$$

$$2NH_3+2(1/2)O_2=NO+3H_2O \quad (3)$$

A thermodynamic description of these processes is given in Table 2.1, and all of these reactions are practically irreversible[21].

Table 1.

Thermodynamic indicators of ammonia oxidation process.

The reaction equation	ΔH^0_{298}, kcal/mol	ΔG^0_{298}, kcal/mol
$2NH_3+1(1/2)O_2=N_2+3H_2O$	-151,4	-156,1
$2NH_3+2O_2=N_2O+3H_2O$	-131,9	-131,2
$2NH_3+2(1/2)O_2=NO+3H_2O$	-108,2	-114,7

There is a possibility that the dinitrogen monoxide formed based on the above reaction (2) decomposes into N_2 and O_2 molecules on the GSM surface at high temperature or combines with NH_3 to form N_2 and H_2O molecules:

$N_2O \rightarrow N_2 + 1/2\ O_2$

$N_2O + NH_3 \rightarrow N_2 + H_2O$

One of the well-known methods of determining the direction of the reaction is based on determining the composition of the resulting products. In the course of the research, the amount of the initial products (NH_3) and the resulting components such as N_2, N_2O and NO in the presence of ammonia-detecting semiconductor sensor gas-sensitive material (10% Fe_2O_3+90%TiO_2) components of ammonia in directions (1)-(3) was measured by gas chromatography, photocolorimetric and potentiometric controlled using methods.

Gas chromatographic analysis of the mixture of N_2, O_2, NH_3, N_2O and NO was carried out on a "crystal-500" type instrument equipped with a heat transfer detector (catarometer). The amount of NH_3, N_2O and NO in the reaction product was determined by additional photocolorimetric method [22]. Potentiometric determination of ammonia is based on dissolving a known volume of the reaction product in a 0.01 N sulfuric acid solution, potentiometric titration of the resulting solution with a 0.01 N alkali solution. The amount of nitrogen in the product of the reaction carried out at low temperatures, which is presented in Table 2, was determined using the difference between the concentration of ammonia before and after the

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reaction. The amount of nitrogen in the reaction product at high temperatures, which is accompanied by the formation of nitrogen oxides as a result of the reaction, was determined using the difference between the amount of ammonia involved in the reaction and the amount of ammonia spent on the formation of nitrogen oxides.

Table 2.

Composition of oxidation products of ammonia in the presence of 90%TiO_2+10%Fe_2O_3: the amount of NH_3 in the mixture is 2.0%, O_2:NH_3=10, the volume of the catalyst is 1 ml, the gas flow rate is 0.5 l/min).

	Temperature, °C	Reacted NH_3,%	Produc	
			N_2	
1	250	87	87	
2	300	100	100	
3	350	100	100	
4	375	100	91	
5	400	100	77	
6	475	100	19	
7	500	100	8	

We can see from the table that in the temperature range of 150-350^{0}C, the composition of the product of the catalytic oxidation process of ammonia in the presence of 90%TiO_2+10%Fe_2O_3 consists of only nitrogen, at 375-450^{0}C it consists of a mixture of N_2 and N_2O, and above 450^{0}C it consists of N_2 and N_2O and NO. Also, it can be observed from the table that the change of the amount of nitrogen in the reaction product in the studied temperature range (150-500^{0}C) passes through the maximum corresponding to 300-350^{0}C, and above 450^{0}C there is a sharp increase in the amount of N_2O. The results of the analysis showed that the oxidation process of ammonia in the presence of 90%TiO_2+10%Fe_2O_3, used as a gas-sensitive material, proceeds according to the 1st reaction, which proceeds only with the formation of nitrogen in the studied range of 150-350^{0}C.

Thus, the analysis of the composition of the products of the oxidation process showed that at low temperatures, the reaction product is nitrogen, and with increasing temperature, first N_2O is formed, then NO. The conducted studies showed that it is desirable to carry out the process of determining ammonia from gas mixtures using a semiconductor sensor developed on the basis of a gas-sensitive nanocomposite with a composition of 90%TiO_2+10%Fe_2O_3 at a temperature of 300-350^{0}C on the GSM surface.

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b). The mechanism and kinetics of the oxidation process of hydrogen sulfide in the presence of 90% WO_3+10% CuO. To date, there is no unified view of the mechanism of H_2S oxidation. Mechanisms of H_2S oxidation in the presence of various metal oxides were proposed, which included: 1-oxygen dissociative adsorption, 2-H_2S adsorption, and 3-H_2S and O_2 adsorptions. However, the studies focused on studying the laws of the oxidation process in the presence of semiconducting metal oxides used as chemical sensor gas-sensitive material are limited. Therefore, it is important to study the mechanism and kinetics of the catalytic oxidation process of H_2S in the presence of 90%WO_3+10%CuO, which allows to improve the parameters of the sensor.

The process of oxidation of H_2S with air oxygen in the presence of gas-sensitive material components (90%WO_3+10%CuO) for the detection of hydrogen sulfide in the semiconductor sensor as observed in the researches and literature data S and H_2O; S can go in the following three directions with the formation of SO_2 and H_2O and SO_2 and H_2O.

$$2H_2S + O_2 = 2S + 2H_2O \qquad (4).$$

$$2H_2S + 2O_2 = S + SO_2 + 2H_2O \qquad (5).$$

$$2H_2S + 3O_2 = 2SO_2 + 2H_2O \qquad (6).$$

In order to study the mechanism and kinetics of the oxidation of hydrogen sulfide on the surface of a gas-sensitive material, the reaction product obtained under different conditions and the composition of the initial product were determined by gas chromatographic, photocolorometric potentiometric and chemical-titrimetric (iodometric) methods under the following conditions [22, 23]. Gas chromatographic analysis of the reaction product was carried out on a computerized instrument of the "Krystal-500" type. In the experiments, the amount of C produced by reactions 4 and 5 was calculated based on the following equations.

$$X_S =(C^0_{H2S} - C^1_{H2S}) *100/ C^0_{H2S} \qquad (7)$$

$$X_S = C^0_{H2S} - (C_{SO2}+ C^1_{H2S}) *100/ C^0_{H2S} \qquad (8).$$

Here, X_S is the amount of hydrogen sulfide converted into sulfur according to reaction equation 4 or 5, %. The amount of C^0_{H2S}-hydrogen sulfide in the initial mixture (before the reaction), %, C^1_{H2S} - the amount of hydrogen sulfide in the mixture after the reaction, %, the amount of C_{SO2} SO_2 in the reaction product. During the research, the effect of temperature on the speed of the oxidation process and the direction of the reaction was studied. The experiments were carried out in the temperature range of 200-400^{0}C, gas flow rate - 5l/min, and H_2S concentration in the gas-air mixture at a constant 3%.

The results of the analysis of the composition of the product of the oxidation process with the presence of 90%WO_3+10%CuO of hydrogen sulfide at different temperatures are presented in Table 3.

Table 3

The composition of the product of the oxidation process of H_2S at different temperatures (catalyst 90%WO_3+10%CuO, n=5, p=0.95, gas flow rate - 5l/min., O_2/H_2S=7)

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T/	Temperature, ^0C	Content of H_2S, %		H_2S conversion, %	Product composition, %	
		Before the reaction	After the reaction		S	SO_2
1	200	3,0	1,6	46,7	46,7	0,0
2	240	3,0	0,8	73,4	73,4	0,0
3	260	3,0	0,3	90,0	83,0	7,0
4	300	3,0	0,0	100	60,3	39,7
5	320	3,0	0,0	100	21,8	78,2
6	340	3,0	0,0	100	0,0	100
7	400	3,0	0,0	100	0,0	100

From the results presented in Table 3, we see that the product of the oxidation process of hydrogen sulfide in the temperature range of 200-240^{0}C in the presence of a semiconductor oxide mixture containing 90%WO_3+10%CuO consists only of sulfur. So, in this range of temperature, the oxidation process of H_2S goes according to the reaction equation (4). From the table, we see that in the range of 200-240^{0}C, the amount of sulfur in the reaction product increases to 46.7-73.4%. Hence, the reaction is completely shifted towards sulfur formation in this range. The maximum value (89.5%) of sulfur in the presence of used metal oxides corresponds to 280^{0}C. In the temperature range of 280-320^{0}C, the presence of sulfite anhydride along with sulfur was found in the product of the oxidation process. So, at temperature values from 260 to 320^{0}C, the process proceeds with simultaneous formation of S and SO_2 according to equation (5). From the table, we can see that the sulfur in the reaction product decreases from 89.5% to 21.8% and the amount of sulfite anhydride increases from 10.5% to 78.2% with the increase in temperature.

From 340 ^{0}C, the reaction product consists only of SO_2 and N_2O. Therefore, in the studied range from 340^{0}C to -400^{0}C, the oxidation of H_2S in the presence of 90%WO_3+10%CuO proceeds with the formation of only SO_2 according to the third equation. As a result of checking the composition of the product of the oxidation process of hydrogen sulfide at different temperatures, the directions of the oxidation reaction of H_2S corresponding to different values of temperature on the surface of the gas-sensitive material containing 90%WO_3+10%CuO in the temperature range of 200-400^{0}C were determined. Accordingly, at temperatures up to 320^{0}C, the sulfur formed over time can cover the surface of the gas-insensitive material and, as a result, cause a decrease in the sensor signal. For this reason, it is advisable to carry out the analysis with the help of the H_2S detection sensor in the presence of 90%WO_3+10%CuO at the temperature of the GSM surface at values higher than 320^{0}C.

c). Kinetics and mechanism of CH_4 oxidation process in the presence of gas-sensitive material (90%ZnO+10%CoO). The semiconductor sensor is based on the oxidation of methane in the presence of metal oxides, which are

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the components of the gas-sensitive material, with the formation of SO_2 and water, which is the most thermodynamically favorable [24]. This reaction has a large heat effect ($-\Delta H^0_{298}$=191b8 kcal/mol) and is practically irreversible. In cases where oxygen and temperature are not sufficient for the oxidation (combustion) process, the process proceeds in two directions with simultaneous formation of CO2 and water and CO and water. There is also a possibility of formation of HCHO, CH_3OH and HCOOH during catalytic oxidation of methane on GSM surface. Summarizing the information available in the literature [25-27], the following directions can be distinguished from the processes of catalytic oxidation of methane with air oxygen in the presence of n- and p-type semiconducting metal oxides:

1.$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$

2.$CH_4 + 3/2O_2 \rightarrow CO + 2H_2O$

3.$4CH_4 + 7O_2 \rightarrow 8H_2O + 2CO_2 + 2CO$

4.$CH_4 + O_2 \rightarrow HCHO + H_2O$

5.$2CH_4 + O_2 \rightarrow 2CH_3OH$

6.$2CH_4 + 3O_2 \rightarrow 2H_2O + 2HCOOH$

During the conducted research, the gas-insensitive material of the semiconductor sensor was studied with the presence of zinc and cobalt oxides (90%ZnO+10%CoO) of methane oxidation. The conversion of methane with selected oxides was carried out in the temperature range of 200-600°C, in a continuous gas flow of the gas mixture at a speed of 0.5 l/min, in a reactor made of quartz glass with a diameter of 1 cm.

In the experiments, the amount of CH_4, O_2, CO_2, CO, H_2O, HCHO, CH_3OH and HCOOH in the reaction product was checked using modern optical, electrochemical and gas chromatographic methods. Determining the amount of the above-mentioned components from the composition of the reaction products carried out under different conditions is important for finding the optimal conditions of the oxidation process on the surface of the gas-inert material and allows determining the direction of the reaction under different conditions. The results of the analysis revealed the absence of HCHO, CH_3OH and HCOOH in the composition of methane oxidation products under the studied conditions (at atmospheric pressure, temperature range of 200-600^{0}C, O_2/CH_4=7 ratio and gas flow rate values of 0.5 l/min) with the participation of selected GSM components. . For this reason, the dynamics of changes in the amount of reaction products CO, CO_2 and CH_4, which did not participate in the reaction, in the reaction mixture were monitored in order to control the degree of methane oxidation and the direction of the process under different conditions. In parallel experiments, the amount of CO_2 from the composition of the reaction product was additionally determined before and after the reaction by titrating a solution of absorbed alkali (0.1 n sodium hydroxide) with 0.1 M hydrochloric acid. The formation of CO and CO_2 is a process with a large amount of heat release corresponding to the following reactions.

$$CH_4 + 0.5O_2 \leftrightarrow CO + 2H_2 + 35,6\ kJ/mol.$$

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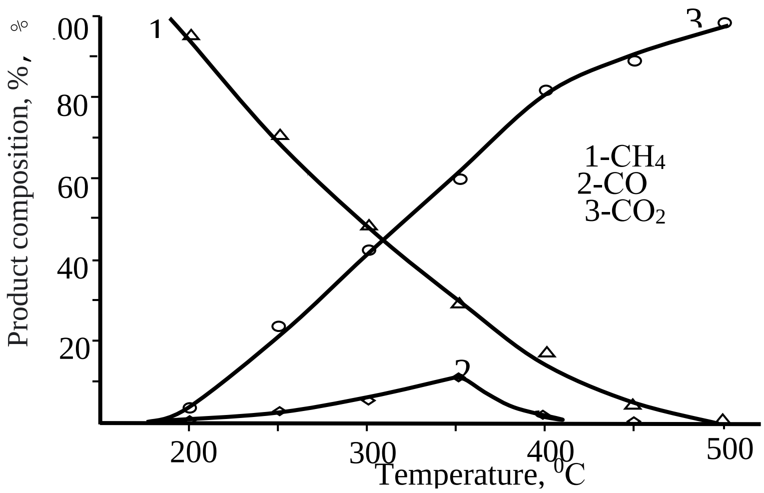
$$CH_4 + 2O_2 \leftrightarrow CO_2 + 2H_2O + 890\ kJ/mol.$$

Therefore, natural gas, the main composition of which is methane, is used as a heat source. This in turn requires the semiconductor sensor to be developed to be able to detect natural gas. Due to the fact that the separation of the products of the oxidation process of methane by the gas chromatographic method in 1 column at the same time caused certain difficulties, the analyzes were carried out in the chromatograph "Gazokhrom-3101" equipped with three columns specially for the control of the composition of the combustion processes.

The graph of the temperature dependence of the composition of the methane conversion process product is presented in Fig. 1.2.

From Figure 3, we see that the amount of methane in the reaction product has decreased from 95% to 6% in the temperature range of 200-450^{0}C. A 100% conversion of CH_4 in the presence of a selected catalyst (the state where the product content is equal to 0) corresponds to a temperature value of 490^{0}C.

Figure 1.2. Dependence of the composition of the product of the



methane oxidation process on the reaction temperature.

The value of CO and CO_2 in the composition of the reaction product also changes with the change in temperature. The composition of the product in 200^{0}C is: 95% CH_4, 4% CO_2 and 1% CO. In 350^{0}C, this reading is: 28% CH_4, 61% CO_2, and 11% CO. The amount of CO in the reaction product passes through a maximum of 11% at 350^{0}C. At further temperatures, we see an increase in CO_2 in the reaction product, and a decrease in CO. As a result, the composition of the product in 400^{0}C is: 16% CH_4, 81% CO_2 and 3% CO. The amount of CO_2 in the product reaches its maximum (100%) point at a temperature of 490-500^{0}C. The results presented in Fig. 3 showed that the oxidation mechanism of CH_4 in the presence of gas-insensitive material components of the methane-detecting semiconductor sensor goes in several directions according to the temperature on the surface of 90%ZnO+10%CoO. The presence of CO and CO_2 in the product in the temperature range of 200-400^{0}C indicates that the 1st and 2nd reactions take place in parallel on the GSM surface in this range. In this case, the rate of reaction 2, which leads to the

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formation of CO, passes through a maximum corresponding to 400^0C. At temperatures above 400^0C, the carbon dioxide formed on the surface of the catalyst is completely oxidized to CO_2, and the mechanism of the methane oxidation process is in the form of reaction 1. The results of the research show that the complete oxidation of the detectable component to CO_2 in the process of detecting methane from gas mixtures using a sensor developed on the basis of a gas-sensitive material with a composition of 90%ZnO+10%CoO corresponds to temperatures higher than 450^0C.

Conclusions.

In the conducted studies, the laws of oxidation of H_2S, NH_3 and CH_4 with the presence of metal oxides were studied, the composition of reaction products with the presence of highly active catalytic systems containing 10%Fe_2O_3-90%TiO_2, 10%CuO-90%WO_3, 10%CoO-90%ZnO was checked and the mechanism of the oxidation process and optimal temperature ranges were proposed.

The catalytic properties of metal oxides in the oxidation process were studied, and based on them, the composition of selective catalysts for semiconductor sensors detecting NH_3, H_2S and CH_4 was found. On the basis of the obtained materials, gas-inert materials for competitive, selective semiconductor sensors of ammonia, hydrogen sulfide and methane were created and their performance was determined.

The results obtained in this section will be used in further studies in the research of sensitive elements of gas sensors and in the development of models of gas-sensing elements for monitoring of H_2S, NH_3 and CH_4 using composite materials based on SiO_2/WO_3+CuO, SiO_2/ZnO+CoO and SiO_2/TiO_2 +Fe_2O_3.

The use of this sensor is of particular importance in improving the metrological and operational indicators of the natural gas alarm and reducing its detection error.

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