UDC 66.074.3:661.98 DOI https://doi.org/10.35546/kntu2078-4481.2024.4.9

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OXIDATION OF NITROGEN OXIDES IN THE PRESENCE OF LOW-TEMPERATURE CATALYSTS

The paper presents the results of the study of the properties of some natural and artificial materials, as well as well-known catalysts in chemical synthesis for the use of increasing the rate of the chemical reaction of nitrogen (II) oxide oxidation. Platinum-based catalysts are very expensive and prone to mechanical erosion, so studying the catalytic properties of new materials is relevant today. The effectiveness of the catalyst was evaluated by comparing the concentration of nitric acid in the case of using a catalyst and in its absence, when oxidation occurred only in the gas phase. The work studied the influence of such factors as the nature of the catalyst, concentration of nitrogen (II) oxide, oxygen concentration, temperature and contact time on the rate of oxidation of nitrogen (II) oxide. It was found that hopkalite, carboalumogel, silica gel, coal, and coke are the most effective of the twelve samples of catalysts. The effect of temperature in the range of 25-80°C on the catalytic properties of materials was studied. At a temperature of 50°C, hopkalite exhibits significant catalytic activity. It was determined that the method of dosing nitrogen (IV) oxide in order to accelerate the degree of oxidation of nitrogen (II) oxide is possible for systems with a low gas concentration (0.1-0.5%). The work describes the use of oxidizing methods of gas purification in industrial atomizing absorbers as a promising method of sanitary purification of gases from nitrogen oxides. Schemes, working principles and the mechanism of chemical processes for sewage treatment plants with two or more stages of absorption are given.

Key words: *oxidation nitrogen (II) oxide, low-temperature catalyst, sanitary purification of gases, industrial absorbers.*

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ОКИСНЕННЯ ОКСИДІВ НІТРОГЕНУ В ПРИСУТНОСТІ НИЗЬКОТЕМПЕРАТУРНИХ КАТАЛІЗАТОРІВ

В роботі приведені результати дослідження властивостей деяких природних і штучних матеріалів, а також добре відомих у хімічному синтезі каталізаторів для застосування підвищення швидкості хімічної реакції окиснення оксиду нітрогену (ІІ). Каталізатори на платиновій основі дуже коштовні та схильні до механічної ерозії, тому вивчення каталітичних властивостей нових матеріалів на сьогодні є актуальним. Ефективність каталізатора оцінювали методом порівняння концентрації нітратної кислоти у випадку із застосуванням каталізатора і при його відсутності, коли окиснення відбувалось тільки в газовій фазі. В роботі вивчали вплив на швидкість окиснення оксиду нітрогену (ІІ) таких чинників, як природа каталізатора, концентрація оксиду нітрогену (ІІ), концентрація кисню, температура і час контакту. Дослідили, що з дванадцяти зразків каталізаторів найбільш ефективними є гопкаліт, карбоалюмогель, силікагель, кісточкове вугілля, кокс. Вивчали вплив на каталітичні властивості матеріалів температури в межах 25-80°С. За температури 50°С виявляється значна каталітична активність гопкаліту. Визначили, що метод дозування оксиду нітрогену (IV) з метою пришвидшення степені окиснення оксиду нітрогену (ІІ) можливий для систем з низькою концентрацією газу (0,1-0,5%). В роботі описано застосування окиснюючих методів очищення газів в промислових розпорошуючих абсорберах в якості перспективного методу санітарного очищення газів від оксидів нітрогену. Наведено схеми, принципи роботи і механізм хімічних процесів для очисних споруд з дво- і більше ступінчастою абсорбцією.

Ключові слова: *окиснення оксиду нітрогену (ІІ), низькотемпературний каталізатор, санітарне очищення газів, промислові абсорбери.*

Statement of the problem

One of the stages of nitric acid production is the oxidation of nitrogen (II) oxide. Nitrous gases obtained at the stage of ammonia oxidation contain nitrogen (II) oxide, nitrogen, oxygen and water vapor [1, 2]. During the oxidation of nitrogen (II) oxide to nitrogen (IV) oxide, three parallel reactions occur:

$$2NO + O_2 \Leftrightarrow 2NO_2, \quad \Delta H = -112.3 \text{ kJ} \tag{1}$$

$$2NO_2 \Leftrightarrow N_2O_4, \quad \Delta H = -57.0 \text{ kJ} \tag{2}$$

$$NO_2 + NO \Leftrightarrow N_2O_3, \quad \Delta H = -40.0 \text{ kJ} \tag{3}$$

All reactions are reversible, occur in a homogeneous system with the release of heat and a decrease in volume. As a result, a decrease in temperature and an increase in pressure shift their equilibrium to the right.

All reactions are reversible, occur in a homogeneous system with the release of heat and a decrease in volume. As a result, a decrease in temperature and an increase in pressure shift their equilibrium to the right.

It is known that the rate of oxidation of nitrogen oxide by gaseous oxygen increases in the presence of certain solid selective catalysts. The greatest activity is exhibited by catalysts prepared on the basis of precious metals [3, 4]. For example, in nitrogen fertilizer plants, metal platinoid systems, consisting mainly of platinum, rhodium, ruthenium and palladium, are usually used as catalysts [1, 5]. Platinum mesh, as catalysts, have a number of disadvantages. Firstly, they are subject to mechanical erosion at the conversion temperature due to mechanical impurities contained in the oxidizing gas and air. Chemical erosion occurs under the influence of the reaction mixture. Secondly, platinum ones mesh and are susceptible to the influence of catalytic poisons, which often leads to irreversible losses of expensive catalyst and a decrease in the yield of nitrogen oxides [6]. The developed platinoid catalysts sufficiently satisfy industrial conditions, however, due to the high cost and shortage of platinoids, especially in Ukraine, continuous research is being conducted to develop new catalyst formulations in two directions: replacing platinoids with cheaper metals and creating competitive non-platinum catalysts based on metal oxides [7, 8, 9]. The catalytic properties of oxides of 3d-transition elements deposited on a fused α- Al_2O_3 (corundum) [10].

The purpose of the study – to study the catalytic properties of a number of natural and artificial materials, known catalysts for increasing the rate of oxidation of nitrogen (II) oxide with their subsequent use in industry.

Presentation of the main research material

It is known that the rate of oxidation of nitrogen oxide by gaseous oxygen increases in the presence of some solid catalysts. The greatest activity is demonstrated by catalysts prepared on the basis of precious metals.

In this work, the catalytic properties of a number of natural and artificial materials and catalysts were investigated with the aim of using them in industry to increase the rate of NO oxidation.

The study was conducted on a setup consisting of a NO oxidation reactor and a nitrogen oxide absorption reactor. The setup is equipped with auxiliary devices and control and measuring instruments for carrying out the process.

Based on the amount of absorbed nitrogen oxide, one can state the degree of NO oxidation, assuming that the degree of absorption is directly proportional to the degree of gas oxidation.

The studies were conducted by comparing with the variant when there was no catalyst in the reactor, and oxidation occurred only in the gas phase.

During the studies, the influence of some factors on the rate of NO oxidation was studied. Such as the nature of the catalyst, nitrogen oxide concentration, oxygen concentration, temperature, contact time. The parameters were varied within the following limits: concentration NO ($C_{NO} = 1 \div 2{,}25\%$); concentration O_2 ($Co_2 = 5 \div 20\%$); contact time ($\tau = 0{,}52 \div 1{,}26s$); temperature t ($26 \div 30°C$); initial oxidation state NO ($d_o = 25\%$).

The following substances were tested as catalysts:

– large-pore and fine-pore silica gel, contact mass, calculated as V_2O_5;
– hopcalite in the form of a mixture of copper oxide and manganese oxide on a ceramic carrier;
– vanadium catalyst (BAS) with a content of 7.5 ÷ 8% vanadium in the dry;
– manganese (IV) oxide MnO_2;
– alumogel;
– carboalumogel;
– catalysts LTK-4;
– GIAP-10;
– coke;
– apricot kernel coal and a number of catalysts obtained by impregnating coke with catalytically active substances.

The results of studies of various catalysts are presented in Table 1.

The presented data indicate that some catalysts are capable of increasing the rate of heterogeneous oxidation reaction by 1.5-2.5 times (these data are somewhat underestimated due to incomplete absorption of oxidized nitrogen oxides). The reaction of heterogeneous catalytic oxidation of nitrogen oxide has a positive temperature coefficient, which is associated with the manifestation of increased catalytic activity of catalysts with increasing temperature. Of all the tested catalysts, the most effective effect on the rate of NO oxidation was exerted by: hopcalite, which made it possible to increase the degree of absorption of nitrogen oxides by 2.51 times. Second place is occupied by carboalumogel – 1.91 times. In third, fourth and fifth places are silica gel by 1.46 times, stone coal by 1.31 times and coke by 1.17 times. The use of these catalysts allowed to increase the degree of nitrogen oxide absorption by 2.51>1.91>1.46>1.31>1.17 times, respectively. Hopcalite turned out to be the most active which is also mainly used as a catalyst for low-temperature oxidation of carbon (II) oxide [11]. Therefore, more complete studies of this catalyst were carried out. Significant catalytic activity of hopcalite manifests itself already at a temperature of 50°C. Increasing the temperature above 80-100°C is impractical due to the large heat costs for heating the gas and its subsequent cooling before absorption. With an increase in the volumetric gas velocity, the amount of oxidized NO decreases, which is associated with a shorter residence time of NO in the catalysis zone. With an increase in the concentration of O_2 and NO, an increase in the oxidation rate is observed.

Table 1

Results of the study of the catalytic activity of catalysts

Name of the catalyst	T°C	Concentration of obtained HNO_3, g/dm^3		Increasing the degree of absorption in the presence of a catalyst
		Without catalyst	In the presence of a catalyst	
Silica gel (КСК)	25	7.35	8.12	1.105
	50	7.93	8.43	1.064
	80	7.86	11.48	1.460
Silica gel (КСМ)	25	7.30	4.53	0.622
	50	6.73	4.92	0.73
	80	6.11	5.92	0.968
V_2O_5	25	6.74	5.48	0.814
	50	6.35	5.48	0.873
	80	5.53	5.48	0.990
Alumogel	25	8.85	6.65	0.765
	50	7.73	7.55	0.977
	80	7.30	9.55	1.31
Carboalumogel	25	9.0	9.63	1.05
	50	8.80	10.3	1.17
	80	8.37	16.0	1.91
Hopcalite	25	9.17	9.63	1.05
	50	9.00	14.3	1.59
	80	8.37	21.0	2.51
MnO_2	25	7.70	8.08	1.05
	50	7.40	7.62	1.03
	80	7.35	7.25	0.986
Coke 1	25	6.55	6.55	1.0
	50	6.36	6.73	1.06
	80	6.05	7.05	1.17
Coke 2	25	9.56	8.76	0.917
	50	8.89	8.30	0.934
	80	8.43	8.05	0.955
Coke impregnated with salts Mn и Cu	25	6.30	6.67	1.06
	50	6.11	6.35	1.04
	80	5.22	6.23	1.13
GIAP-10	25	9.17	7.87	0.859
	50	8.95	8.75	0.989
	80	8.55	8.81	1.030
Apricot kernel charcoal	25	7.68	6.66	0.87
	50	5.67	7.16	1.27
	80	5.67	7.41	1.31

Increasing the degree of NO oxidation by adding concentrated nitrogen dioxide to the gas

Along with the intensification of the NO oxidation process, which is necessary to increase the rate of nitrogen oxide absorption, a similar effect can be achieved by dosing the gas with an appropriate amount of NO_2.

The trioxide obtained in this way can be successfully absorbed using one of the known methods.

To increase the degree of NO oxidation by α_1 до α_2 nitrogen dioxide must be added to the gas mixture, the amount of which can be determined from the equation

$$\Delta V_{NO_2} = \frac{Vr \cdot Cr}{100}\left(\frac{\alpha_2 - \alpha_1}{1-\alpha_2}\right), \text{ m}^3/\text{h} \qquad (1)$$

where, Vr и Cr – respectively the quantity (m^3/h) and concentration of the initial gas mixture (%).

The concentration of gas after dosing NO_2 and depending on the required degree of gas oxidation will be

$$C'r = Cr\left(\frac{1-\alpha_1}{1-\alpha_2}\right) \qquad (2)$$

The NO_2 dosing method for increasing the NO oxidation degree is only acceptable for systems with a low initial gas concentration (0.1-0.5%) and a low NO oxidation degree. In this case, the NO_2 dosing method for obtaining a reactive mixture N_2O_3 is more economically advantageous than the method of oxidation of NO to N_2O_3 in oxidizing volumes.

Introduction of oxidizing methods of sanitary gas purification from nitrogen oxides in industry

Of the new methods of sanitary gas purification from nitrogen oxides developed, the most promising are widely used in industry. Oxidizing methods of gas purification in hollow spray absorbers have proven themselves well.

The degree of purification of 0.1-0.5% gas with an oxidation degree of 0.2-0.3 is 80-85%. A higher degree of gas purification from nitrogen oxides is achieved in devices with lower productivity (Fig. 1).

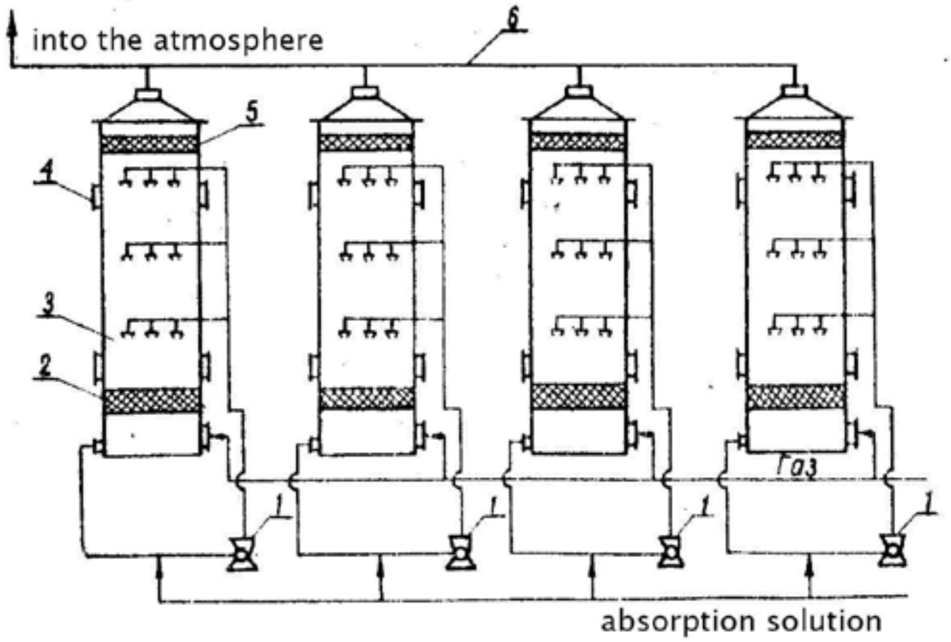

Fig. 1. Scheme of industrial installation for gas cleaning in hollow devices: 1 – pumps; 2 – distribution screen, 3 – nozzles; 4 – manhole; 5 – drip separator; 8 – discharge manifold

To purify 75 thousand m^3/hour of nitrous gas, one of the enterprises has 4 hollow spray towers installed in parallel. Impact spray nozzles are arranged in four tiers along the height of each tower. With a spray density of 6 m^3/m^2 hour and a volumetric gas velocity of 150-200 h^{-1}, the degree of purification of highly oxidized gas with a Na_2CO_3 solution is 96-88%. Long-term operation of treatment facilities of this design has confirmed the reliability and high efficiency of hollow spray devices.

At some enterprises that emit gases with a high content of nitrogen oxides into the atmosphere, the developed treatment facilities with two-stage absorption are successfully used. Such installations have proven themselves well in operation in a variable mode (in terms of the amount and composition of emitted gases), as well as with periodic emissions of exhaust gases. At one of the operating installations (Fig. 2), a NaOH solution is used as the first stage absorber, and at the second stage, an aqueous ammonia solution containing 1.5-2.0% ammonia is used for additional purification.

The gases to be purified are characterized by inconstant composition, temperature and pressure. Therefore, the system provides for preliminary gas preparation. The gases supplied for purification pass through valves 1, 2 and are directed to the common manifold 3. After passing the pressure regulator 4, the gas is directed through the main valve 6 along the gas duct 14 to the first-stage absorber 12. If the temperature of the incoming gas is below 0°C, it is heated to 10-20°C in the heat exchanger 5, into the inter-pipe space of which steam is supplied. The pressure regulator 4 automatically, regardless of the pressure in the common manifold, ensures a certain uniform gas flow during the entire period of its supply to the absorber. If the gas has a temperature of 250-350°C, it, as a rule, contains a large amount of NO and therefore, in addition to cooling, requires additional oxidation. For this purpose, the gas is directed through valve 9 to heat exchanger 8, where it is pre-cooled to a temperature of 20-30°C and then through pressure regulator 7 is directed to post-oxidizer 13. Air or oxygen is supplied here by high-pressure fan 10 through valve 11 in the amount necessary for oxidation of nitrogen oxide.

The volume of post-oxidizer is calculated in such a way that the gas passing through it has time to oxidize by at least 50%, which is necessary for successful gas purification with alkaline solutions. The gas prepared for purification enters first-stage absorber 12 through gas duct 14. This absorber is made in the form of a hollow stainless steel cylinder. The first stage absorber is irrigated with an 8-10% aqueous solution of NaOH from tank 17, the second stage – with a 1.5-2.0% solution of NH_4OH using centrifugal pumps 15. The preparation of working solutions of NaOH and NH_4OH is carried out in tanks 17, 16 and 19.

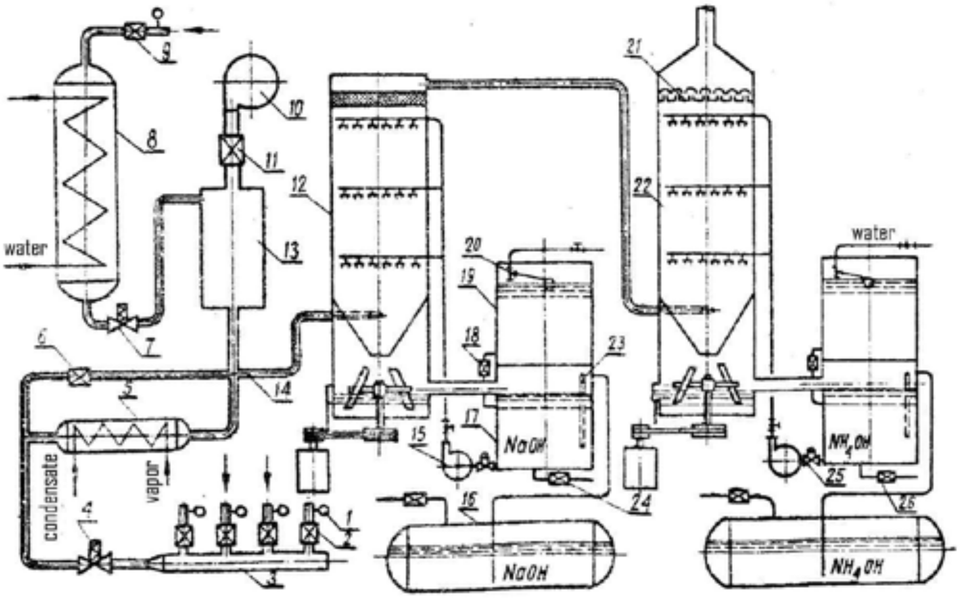

Fig. 2. Diagram of a two-stage industrial installation for cleaning gases from nitrogen oxides

The operation of the unit is fully automated. The level of solutions in the preparation tanks is maintained automatically by a level gauge 23, floats 20 and regulators 18. The waste liquid is drained through valves 24, 26, the liquid supply to the absorbers is adjusted by valve 25. In order to prevent splash entrainment, a drip separator 21 is provided on the second-stage absorber 22. The system capacity is 2.5-3.0 m^3/s. The nitrogen oxide content in the feed gas is 0.5-12%. The first stage provides a gas purification degree of 95-99%, reducing its concentration to 0.12-0.63%. After the second stage, the purification degree is 99.9-100%. The use of ammonia solution as an absorbent in the second stage leads to the formation of an aerosol of nitrite and ammonium nitrate in the exhaust gases, which decomposes into nitrogen and water in the air atmosphere. At low ambient temperatures, a thermal reactor must be used to decompose aerosols, in which at $t=200^oC$ the aerosols completely decompose into nitrogen and water. With an initial concentration of nitrogen oxides in the gas of up to 7%, the sanitary standard can be achieved in the first-stage absorber. Installations of this design are accepted as standard and are successfully operated at a number of related enterprises. Hollow spray absorbers are widely used in the sulfuric acid industry to capture nitrogen oxides in the absorption zone. The oxidative method, where the oxidation and absorption of oxides was carried out using ammonium carbonate solutions of hydrogen peroxide, also passed the stage of semi-industrial tests. The pilot plant, the general appearance of which is shown in Fig. 3, was mounted in the hydroxylamine sulfate shop of one of the chemical plants. The basis of the plant consists of three absorption devices 6 with foam phase contact. The devices are made of Czech glass of standard dimensions (diameter – 300 mm, height – 1.5 m). In the lower part of each device a grid with a live cross-section of 11% (diameter – 2 mm, thickness – 6 mm) is installed.

A drain threshold 5 is mounted in the grate, the lower end of which is connected to the drain tank 2 via a seal 4 and a foam suppressor 3. To drain the failing liquid, the lower part of the absorber is connected to the drain tank via a valve 11. The working solution was circulated in the absorbers using pumps 12. The solution supply for irrigation was regulated using a valve 13 and a rotameter 14. The ammonium carbonate solution and hydrogen peroxide were supplied from pressure tanks 14 and 8 via rotameters 15 and 7. The installation is equipped with gas sampling points 9, 10 and liquid 1 after each absorber. Gas and liquid analysis were performed twice per shift with 3-5-fold duplication.

The gas consumption in all studies was 76 m^3/h, the consumption of fresh solution containing 300 g/dm^3 of ammonium carbonate was 20 dm^3/h with an irrigation rate of 6, which provided an irrigation density of 1.6 $m^3/m^2 \cdot h$. The foam height in each absorber was 200 mm. The operation of the installation in this mode was characterized by the data given in Table 2.

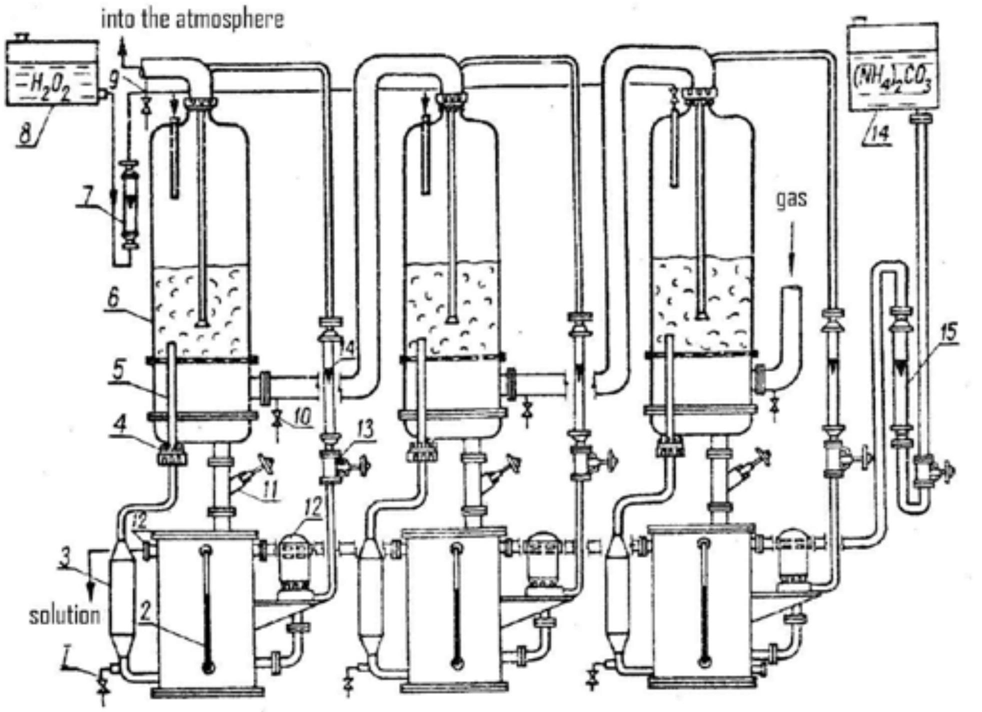

Fig. 3. Scheme of a pilot plant for cleaning gases from nitrogen oxides in the production of ammonium nitrite

Table 2

Pilot unit operating mode

Parameter	Sampling points			
	At the entrance	After 1 absorber	After 2 absorber	After 3 absorber
Concentration of nitrogen oxides, % (vol)	0.213	0.145	0.102	0.075
Degree of purification	-	30	52	64.8

Considering that the spent solution contained some amount of free hydrogen peroxide in addition to nitrite-nitrate mixtures, and this led to its direct loss. Further studies were carried out in a continuous mode for gas and periodic for liquid, i.e. according to a circulation scheme similar to the operation of alkaline absorption departments of nitric acid shops. In this mode, fresh solution was supplied to the unit and spent solution was drained from the system once every two shifts. Hydrogen peroxide was supplied during the entire period of operation directly to each absorber and stopped 1 hour before the solution was released from the system. Such supply allowed for more complete processing of the working solution and the release of alkali from the system that did not contain free hydrogen peroxide. This significantly reduced the consumption of H_2O_2. Long-term operation of the unit has shown that the optimal conditions for its operation are: linear gas velocity in the free section of the apparatus – 0.5 m/s; ammonium carbonate concentration at the beginning of the process – 100÷120 g/dm^3; ammonium carbonate concentration at the end – 10÷15 g/dm^3; irrigation density – 1.5÷2.0 m^3/m^2·h; foam layer height on the shelf – 200 mm. Spent solution from the system had the following composition, g/dm^3: ammonium carbonate – 20; ammonium nitrite – 63, ammonium nitrate – 82; free ammonia – 1.2.

According to the reaction between NO and H_2O_2, the maximum theoretical consumption of hydrogen peroxide per 1000 m^3 of gas is 1.2 kg, assuming that the initial concentration of nitrogen oxides is 0.2%, and the degree of NO oxidation is 20%, and that the nitrogen dioxide present in the gas at the start of the reaction reacts without the consumption of H_2O_2. Considering that part of the hydrogen peroxide is inevitably spent on the oxidation of NO to NO_2 (which is evident from the composition of the resulting alkalis), the actual consumption of hydrogen peroxide should not exceed 2 kg per 1000 m^3 of gas. During the study on the pilot plant, the consumption of hydrogen peroxide was 4 kg per 1000 m^3 of gas, i.e. it exceeded the calculated value by two times. Turbulence of the system by improving the design of the plates and increasing the gas velocity in the apparatus did not lead to a decrease in the consumption of hydrogen peroxide, which indicates that the process occurs in the kinetic region. Under these conditions, an increase in the rate of oxidation and absorption of nitrogen oxides can be achieved by controlling the process of decomposition of hydrogen peroxide. The greatest stability of hydrogen peroxide is observed in an acidic medium at pH = 1÷3. In a neutral medium (pH = 7), it has average stability, and at pH = 11.5÷13, its intensive decomposition is observed. Excessively rapid decomposition of hydrogen peroxide does not allow the active oxygen released in this case to be fully utilized. As studies have shown, the most favorable conditions for the oxidation of nitrogen oxide with hydrogen peroxide solutions are pH = 10÷11 and t = 30°C. Maintaining the alkalinity of the medium at a level (pH = 10÷11), the consumption of hydrogen peroxide was reduced from 4 to 2.5 kg per 1000 m^3 of gas. The degree of gas purification on three shelves reached 75÷80%, the other conditions of the experiments remained unchanged. The residual content of nitrogen oxides in the exhaust gases was 0.04%, which allows this method to be used for systems emitting 20÷30 thousand m^3/hour of gas with an initial content of nitrogen oxides up to 0.2%. For systems with higher productivity, it is necessary to increase the number of absorption stages.

Conclusions

1. The most effective catalysts for the oxidation of nitrogen (II) oxide were: hopkalite, carboaluminum gel, silica gel, coal, and coke.
2. When studying the influence of temperature, the greatest catalytic activity of hopcalite was found at a temperature of 50°C.
3. The application of oxidizing methods of gas purification in industrial atomizing absorbers as an effective method of sanitary purification of gases from nitrogen oxides is described.

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