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EFFICIENCY OF LIQUID OXIDIZERS FOR PURIFICATION OF INDUSTRIAL WASTE GASES FROM NITROGEN OXIDES

Nitrogen oxides are among the most common and dangerous air pollutants on our planet. Exhaust gases, which include nitrogen oxides, are produced in a number of chemical industry productions, in oil refining processes, during the burning of various types of fuel at high temperatures. The work describes the study of the effectiveness of liquid oxidizers for the intensification of the processes of cleaning industrial gases from nitrogen oxides. H_2O_2 , $KMnO_4$, $KBrO_3$, $K_2Cr_2O_7$, Na_2CrO_4 , $(NH_4)_2Cr_2O_7$, $NaOCl$, which are of theoretical and practical interest, were studied as oxidants. To establish the principle possibility of oxidation of nitrogen oxide (II) by oxidants, thermodynamic parameters were calculated. Determination of the rate of oxidation and absorption of nitrogen oxides was carried out on an experimental device. The effect of the concentration of oxidants, temperature, and pH on oxidation processes was studied in the paper. It was found that increasing the concentration of the investigated oxidants accelerates the oxidation and absorption reactions of nitrogen oxide (II). It was established that an increase in temperature accelerates the oxidation processes in the liquid phase in contrast to oxidation in the gas phase. Aqueous solutions of potassium bromate, hydrogen peroxide, and potassium permanganate were found to be the most active of the investigated oxidants in relation to nitrogen oxide (II). Research was conducted by the method of full factorial experiment. As a result and processing of experimental data received kinetic equations of nitrogen oxide (II) oxidation liquid oxidizers depending on the concentration of liquid oxidizers, temperatures, etc and pH of the environment.

Key words: purification of waste gases, oxidation of nitrogen oxides, liquid oxidizers, kinetics of oxidation.

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ЕФЕКТИВНІСТЬ РІДКИХ ОКИСНИКІВ ДЛЯ ОЧИЩЕННЯ ПРОМИСЛОВИХ ВІДХІДНИХ ГАЗІВ ВІД ОКСИДІВ НІТРОГЕНУ

Оксиди нітрогену відносяться до найпоширеніших і небезпечних забруднювачів повітря на нашій планеті. Відхідні гази, до складу яких входять оксиди нітрогену, утворюються у ряді виробництв хімічної промисловості, в процесах нафтопереробки, при спалюванні різних видів палива при великих температурах. Окисні методи санітарного очищення газів від оксидів азоту засновані на попередньому окисненні оксиду азоту (II) з подальшим поглинанням оксидів азоту (IV) та (III) різними поглиначами. В роботі описано дослідження ефективності рідких окисників для інтенсифікації процесів очищення промислових газів від оксидів нітрогену. В якості окисників досліджували H_2O_2 , $KMnO_4$, $KBrO_3$, $K_2Cr_2O_7$, Na_2CrO_4 , $(NH_4)_2Cr_2O_7$, $NaOCl$, які представляють теоретичний і практичний інтерес. Для встановлення принципової можливості окиснення оксиду нітрогену (II) рідкими окисниками проведено розрахунок термодинамічних параметрів. Визначення швидкості окиснення і абсорбції оксидів нітрогену здійснювалося на експериментальному пристрої. В роботі досліджено вплив концентрації окисників, температури і рН на процеси окиснення. Виявлено, що збільшення концентрації досліджуваних окисників пришвидшує реакції окиснення і абсорбції оксиду нітрогену (II). Встановлено, що збільшення температури пришвидшує процеси окиснення в рідкій фазі на відміну від окиснення в газовій фазі. Найбільш активними з досліджуваних окисників по відношенню до оксиду нітрогену (II) виявили водні розчини калій бромату, гідроген пероксиду та калій перманганату. В результаті аналізу експериментальних даних отримані кінетичні рівняння окиснення оксиду нітрогену (II) рідкими окисниками в залежності від концентрації рідких окисників, температури і рН середовища.

Ключові слова: очищення відхідних газів, окиснення оксидів нітрогену, рідкі окиснювачі, кінетика окиснення.

Statement of the problem

The main sources of atmospheric pollution are: natural, artificial, mechanical and chemical pollutants. Chemical pollutants include dusty or gaseous substances capable of chemical reactions. Pollution of the Earth's atmosphere or pollution occurs when harmful or excessive amounts of substances, including gases (such as carbon dioxide, carbon monoxide, sulfur dioxide, nitrogen oxides, methane, and chlorofluorocarbons) enter the Earth's atmosphere [1]. Air pollution can be caused by both human activity and natural processes. At present, the effect on humans of the majority of harmful substances present in atmospheric air has been studied. On the basis of this, the limit-permissible concentrations (LPC) for a number of the most common pollutants are established [2, 3].

Visual evidence of the influence of atmospheric air pollution on human health is provided by statistical data on mass diseases of the population in large industrial cities and cases of catastrophic consequences caused by atmospheric pollution [4].

Nitrogen oxides occupy a special place among more than four and a half thousand pollutants existing today and included in the database. Nitrogen oxides are among the most widespread and dangerous air pollutants on our planet. Exhaust gases, which include nitrogen oxides, are formed in a number of chemical industries, in oil refining processes, when burning different types of fuel at high temperatures.

Analysis of the latest research and publications

Sources of emissions of nitrogen oxides into the atmosphere differ in the amount of exhaust gases, the content of nitrogen oxides and other impurities in them, the degree of oxidation of nitrogen oxide (II) NO, temperature, pressure, etc. The authors implemented a classification of methods of sanitary cleaning of industrial gases from nitrogen oxides. According to the proposed classification, the developed methods of cleaning gases from nitrogen oxides are divided into four classes: oxidative, reducing, sorption and other methods [5–9]. Each class is divided into four groups, each group combines a number of cleaning methods of the same type.

The advantage of the proposed classification is that all existing and developed methods of sanitary gas purification from nitrogen oxides fit into its scheme. Nitric oxide (II) does not dissolve in liquids, so it is necessary to pre-oxidize it. The resulting products dissolve in liquids, which are later used for cleaning industrial gases.

Absorption methods are a process of selective gas absorption by a liquid. Two phases are involved in absorption processes – liquid and gas. At the same time, the transition of the substance from the gas phase to the liquid phase (during absorption) or, conversely, from the liquid phase to the gas phase (during desorption) occurs. Thus, absorption processes are one of the types of mass transfer.

At the basis of absorption are the diffusion processes of the transition of a substance from the gaseous phase to the liquid through the phase interface. The driving force of absorption is the difference between the initial and equilibrium pressures of the interacting components:

$$\Delta P_{cp} = \frac{(P'_z - P''_z) - (P'_p - P''_p)}{2.3 \lg \frac{P'_z - P'_p}{P''_z - P''_p}}, \tag{1}$$

where P'_z, P''_z partial pressures of the absorbed component in the gas phase at the entrance to the apparatus and at the exit from the apparatus; P'_p, P''_p equilibrium partial pressures of the absorbed component above the liquid.

Absorption occurs only if the partial pressure of the absorbed component in the gas phase is greater than the equilibrium partial pressure of the same component above the given solution. The greater the difference between these pressures, the greater the driving force of the process and the faster absorption occurs.

The limit state of the system is the achievement of equilibrium, that is, a state where only reversible processes can occur in it.

Physical absorption and chemisorption are distinguished. During physical absorption, the gas is held in the liquid only due to mechanical (physical) bonds and it is not accompanied by a chemical reaction, while during chemisorption, a chemical reaction occurs between the components.

Oxidation methods of sanitary cleaning of gases from nitrogen oxides are based on the preliminary oxidation of nitrogen oxide (II) with subsequent absorption of nitrogen oxides (IV) and (III) (NO₂ and N₂O₃) by various absorbers [10]. The stage that limits the overall process is the rate of oxidation of nitrogen oxide (II) to nitrogen oxide (IV). Since this reaction takes place in the gas phase, the absorption apparatus must have a sufficiently large free volume, so the absorption process is carried out in multi-stage packed or plate columns. In experiments on the absorption of nitrogen oxides by various liquid absorbers, the degree of absorption was 80–90%. In the process of absorption, nitrogen oxides are oxidized not only in a gaseous but also in a liquid environment. In [10] studies were directed to the intensification of the process of oxidation of nitrogen oxide (II) in the liquid phase. The work compared the rates of NO oxidation in the gas and liquid phases. The industry uses the method of homogeneous oxidation of NO in the gas phase with the help of oxygen. However, as practice shows, at low concentrations of NO, the rate of homogeneous oxidation of nitrogen oxides is extremely low.

One of the methods of intensification of the process of absorption of nitrogen oxides is the oxidation of nitrogen monoxide by oxidants stronger than oxygen, for example, ozone or an ozone-oxygen mixture [10]. However, the use of ozone is impractical due to the low efficiency and high cost of ozone. Studies with the introduction of ozone into the liquid phase showed that in this case ozone acts not only as an oxidizer, but also as a catalyst, which increases the degree of oxidation of nitrogen oxide (II) and the relative rate of absorption by one and a half times. When nitrogen oxides are absorbed by nitric acid, regardless of its initial concentration, the introduction of a small amount of ozone into the liquid phase increases the speed of the absorption process and the degree of oxidation (Table 1).

Table 1

Effect of ozone in the liquid phase on the oxidation of nitrogen oxide (II)

	The maximum concentration of the resulting HNO ₃ , %	Relative rate of absorption
Without ozone	53	1.0
With ozone in the liquid phase (0.16%)	60	1.5

Intensification of the process of homogeneous oxidation of nitrogen oxides in the liquid phase by gaseous oxidants (oxygen or ozone) is associated with the diffusion of oxidants into the liquid phase. Diffusion of oxygen into the solution is the slowest stage of homogeneous oxidation of nitrogen oxides in the liquid phase, and therefore it controls the entire process.

The rate of diffusion of gaseous oxidizers into the liquid phase depends on many factors: temperature, pressure, concentration of components, turbulence of flows, etc. To increase the rate of homogeneous oxidation of NO in the liquid phase, it is necessary to influence the system by changing one or more of the listed parameters.

The purpose of the work is to study the effectiveness of liquid oxidizers for the intensification of the process of purification of industrial gases from nitrogen oxides.

NO oxidation reaction can be increased if it is carried out in the liquid phase with the help of liquid oxidizers. In this case, the oxidizers are in the liquid phase, and the rate of the NO oxidation reaction will not depend on the rate of diffusion of the oxidizers into the solution. Thus, the slowest stage of the process is eliminated. A number of liquid oxidizing agents are known, which are superior in their activity to oxygen. These include aqueous solutions of H_2O_2 , $KMnO_4$, $KBrO_3$, $K_2Cr_2O_7$, $(NH_4)_2Cr_2O_7$, Na_2CrO_4 , $KClO_4$, $NaClO_2$, $K_2S_2O_3$, $(NH_4)_2S_2O_8$, NH_4ClO_4 , $K_2Cl_2O_7$, $K_2S_2O_7$, HNO_3 . Some of these oxidizers are of theoretical and practical interest for the process of oxidation of nitrogen oxide (II) for the purpose of sanitary gas purification. To study the NO oxidation process, the following liquid oxidizers were taken: H_2O_2 , $KMnO_4$, $KBrO_3$, $K_2Cr_2O_7$, Na_2CrO_4 , $(NH_4)_2Cr_2O_7$, $NaOCl$, HNO_3 , and aqua regia.

To find out the principle possibility of interaction of nitrogen oxides with the listed liquid oxidants, thermodynamic parameters were calculated.

Analysis of the results of thermodynamic calculations of ΔH° and ΔS° indicates the principle possibility of the process of oxidation of nitrogen oxide by liquid oxidizers. The greater the numerical value of the equilibrium constant and the decrease in enthalpy, the more complete the reaction can proceed in the forward direction. Large values of equilibrium constants allow us to conclude that these reactions proceed almost to the end.

The experimental determination of the rate of oxidation and absorption of nitrogen oxides was carried out on a mechanized experimental setup, the scheme of which is shown in Figure 1.

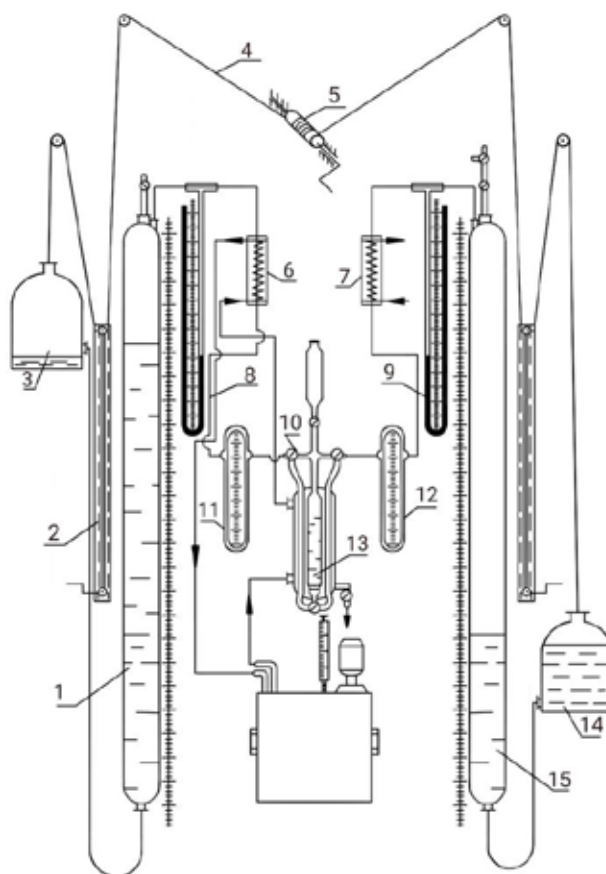


Fig. 1. Scheme of the installation for determining the rate of chemical reaction between gas and liquid:
 1, 15 gas burettes, 2 pressure compensators, 3, 14 equalizing cups, 4, 5 winches, 6, 7 refrigerators,
 8, 9 manometers, 10 three-way faucet, 11, 12 rheometers, 13 absorbing vessel

The installation is mounted on a metal frame and contains a thermostatically controlled absorption vessel 13, two gas burettes 1, 15, two manometers 8, 9, two rheometers 11, 12, and two refrigerators 6 and 7. The gas burettes are equipped with equalizing glasses 3 and 14, operated with the help of a common thrust 4 with a winch 5. To maintain a constant pressure during research, pressure compensators are provided in the installation 2. The installation works as follows. The gas under investigation, in this case nitrogen oxide (II), and the absorbing solution (liquid oxidizer), the amount of which has been precisely measured in advance, are drawn into the gas burette 1 and the absorbing vessel 13, respectively. When the winch 5 is rotated clockwise due to the change liquid level in gas burettes a pressure drop occurs

$$\Delta P = P_1 - (-P_2),$$

where P_1 and P_2 respectively, initial and final pressure in gas burettes.

When ΔP exceeds the resistance of the liquid column in the absorption vessel 13 ($\Delta P > H$), gas from burette 1 will move to burette 15, passing through the absorbing solution in vessel 13. Gas movement will continue until burette 1 is completely filled with water. Having changed the direction of rotation of the winch counterclockwise and turned the three-way valve 10, the gas from the burette 15 through the absorption vessel 13 is again returned to the burette 1. When the gas passes through the absorption vessel, oxidation and absorption of nitrogen oxide (II) occurs, as a result of which the volume of gas in burettes decreases. By changing the volume of gas at any moment of time, which is fixed with the help of instruments, it is possible to determine the amount of oxidized absorbed of nitrogen oxide (II).

The need to create such an installation is due to the fact that in existing devices of this type, gas absorption is accompanied by a simultaneous change in pressure in the system, and the research conditions at the beginning and end of the experiment were not the same. In addition, the gas came into contact with the liquid by shaking the absorbent vessel, which reduced the accuracy and reliability of the experiment. The vacuum formed in the system due to gas absorption is eliminated with the help of pressure compensators 2. By passing gas at a constant rate, which is controlled by rheometers, it is possible to quickly and accurately determine the kinetics of the process and the full absorption capacity of the tested solution.

The rate of oxidation and absorption of nitrogen oxide (II) was determined by the formula:

$$U = \frac{\Delta V_i}{V_p \cdot 22.4 \cdot \tau}, \text{ mol/dm}^3 \cdot \text{s} \quad (2)$$

where ΔV_i – the volume of gas absorbed by the solution during the time interval τ , dm^3 ; V_p - volume of absorbing solution, dm^3 ; τ_i – experiment time, s.

The total absorption capacity of the oxidizer was determined by formula 3:

$$G = \frac{\sum_{i=1}^n \Delta V_i}{V_p}, \text{ dm}^3 / \text{ dm}^3 \quad (3)$$

As the concentration of oxidants increases, the rate of oxidation reaction and absorption of nitrogen oxide (II) increases for all oxidants. Thus, the reaction rate of oxidation and absorption of N by a solution with a mass fraction of hydrogen peroxide of 3% is $200 \cdot 10^{-6} \text{ mol/dm}^3 \cdot \text{s}$, which is 2.14 times more than for a solution with a mass fraction of hydrogen peroxide of 1% ($97 \cdot 10^{-6} \text{ mol/dm}^3 \cdot \text{s}$). A similar increase in the reaction rate is also observed for other oxidants.

Unlike oxidation in the gas phase, the rate of oxidation by liquid oxidants also increases with increasing temperature. The positive temperature coefficient of the reaction is explained by the fact that in the process of oxidation of NO in the liquid phase, there is no intermediate reaction of dimer formation of N_2O_2 , which with increasing temperature shifts to the left, towards the decomposition of N_2O_2 and controls the overall process of oxidation of NO.

An important role is played by the fact that with increasing temperature, the rate of decomposition of liquid oxidants increases with the release of atomic oxygen and active oxygen-containing ions or radicals, which oxidize nitrogen oxide at a high rate. The high oxidizing power of atomic oxygen, oxygen-containing radicals and ions is observed only at the «moment of release», after which they lose their activity. Here it should be taken into account that some oxidation reactions are liquid oxidizers proceed quickly enough even at room temperature. An increase in temperature in this case leads to rapid decomposition of the oxidizer. The released atomic oxygen and other active compounds do not have time to react with nitrogen oxide in the liquid phase, and, passing into the gas phase, lose their activity.

The described regularity applies, first of all, to the oxidizing agent KBrO_3 , where an increase in temperature leads to a decrease in the reaction rate. For all other oxidizers tested, the increase in temperature was invariably accompanied by an increase in the oxidation rate of NO.

On the basis of conducted studies under standard conditions, a range of activity of liquid oxidizers has been established, which decreases in the following sequence:



The reaction rate coefficients for aqueous solutions with a mass fraction of reduced oxidants of 3% respectively have the following values, $\text{mol/l} \cdot \text{s}$:

$$620 \cdot 10^{-6} > 365 \cdot 10^{-6} > 338 \cdot 10^{-6} > 204 \cdot 10^{-6} > 60 \cdot 10^{-6} > 30 \cdot 10^{-6}$$

The given data show that the oxidizing power of various liquid oxidizers differs significantly. An aqueous solution of potassium bromate (KBrO_3) turned out to be the most active of the tested oxidants in relation to NO. Potassium permanganate has somewhat less oxidative activity than KMnO_4 and hydrogen peroxide H_2O_2 . Other liquid oxidizers oxidize NO at a rate insufficient for practical purposes. Such a strong oxidizer as perchloric acid HClO_4 in the given conditions interacts with NO at a very low rate.

Oxidizing potentials of solutions do not always correctly characterize the oxidizing ability of solutions to oxidize NO. Therefore, oxidation potentials cannot serve as a measure of the kinetic ability of liquid oxidants in the reaction with NO. The maximum oxidation potential, for example, is possessed by the H_2O_2 solution, and the maximum rate of oxidation of the solution KBrO_3 . The influence of the concentration, temperature, and pH of the medium on the rate of oxidation by liquid oxidizing agents of nitrogen oxide (II) was studied. Research was conducted by the method of full factorial experiment.

As a result of the processing of experimental data, kinetic equations describing the rate of oxidation of NO by liquid oxidizers depending on the concentration of solutions, temperature, and pH of the medium were obtained. These equations have the form:

– for potassium bromate KBrO_3

$$U_{\text{KBrO}_3} = -[432,65 + 76,25C_{\text{KBrO}_3} - 4,437t + 4,17pH] \cdot 10^{-6} \text{ mol/dm}^3 \cdot \text{s}; \quad (4)$$

– for potassium permanganate KMnO_4

$$U_{\text{KMnO}_4} = [137,5 + 64,75C_{\text{KMnO}_4} + 1,137t + 2,833pH] \cdot 10^{-6} \text{ mol/dm}^3 \cdot \text{s} \quad (5)$$

– for hydrogen peroxide H_2O_2

$$U_{\text{H}_2\text{O}_2} = [29,99 + 176,25C_{\text{H}_2\text{O}_2} + 183,75t + 69,99pH] \cdot 10^{-6} \text{ mol/dm}^3 \cdot \text{s} \quad (6)$$

Conclusions

As a result of research:

- the influence of the concentration, temperature, and pH of the medium of liquid oxidizers on the rate of oxidation of nitrogen oxides was studied;
- the range of activity of liquid oxidizers is established. Oxidizers were selected taking into account the fact that the reaction products must be non-toxic and can be discharged into the sewer without additional neutralization, or can be repeatedly used in the cleaning cycle;
- the most effective oxidizers are defined.

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