

KINETIC PROCESSES IN A PLASMA OF BARRIER DISCHARGE IN ATMOSPHERIC AIR: INFLUENCE OF WATER VAPOR ON BIOLOGICAL ACTIVITY OF THE GENERATED MEDIUM

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Theoretical and experimental investigations of the water vapor influence on the biological activity of the medium generated by barrier discharge in atmospheric air are performed. Numerical calculation of the component composition of the particles generated by barrier discharge in dry and moisture air shows that moistening of air, on the one hand, results in the ozone concentration reduction, but, on the other hand, it results in an increase of the concentration of such a biologically active compound as N_2O_4 and formation of other active compounds – molecules of hydrogen peroxide H_2O_2 , nitric HNO_3 and nitrous HNO_2 acids, and HO_2NO_2 radicals in the discharge. Experiments on the processing of spores have shown that moistening of air leads to an increase of the biological activity of the medium generated by barrier discharge. On basis of experiments and theoretical calculations, one can insist that the rise of the medium activity is connected with the generation of hydric compounds H_2O_2 , HNO_3 , HNO_2 , and HO_2NO_2 .

of any gaseous or vaporous substance, including nontoxic ones (even inert gases). Whereas low-pressure plasma sterilizers already have been commercially available since early 1990's [1], the great attention to the development of low-temperature sterilizers using atmospheric pressure discharges, which are more attractive due to absence of a vacuum system, has been paid for the past years [2 – 4]. However, solving this problem is complicated by the fact that complete investigations of barrier discharge plasma, first of all, that of the component composition of active particles, are not accomplished up to now. The present paper is devoted to the theoretical studies of the component composition of particles, which are generated by a barrier discharge in dry and moist ambient air, and to the experimental investigation of the sterilization efficacy of a barrier discharge depending on air moisture.

Introduction

In the last ten years, gas discharge plasma attains more and more wide applications in biology and medicine for the creation of biomaterials and membranes with pre-defined features (biocompatibility, clot resistance, lowered friction, etc.) and, as well, for cold sterilization of articles made of thermolabile materials. In modern medical practice, the cold sterilization of a wide variety of heat-sensitive instruments and materials is mainly performed by means of toxic gases – pure ethylene oxide or its mixture with fluorochlorocarbons. This sterilization technique requires a long (up to 24 hours) aeration process and, which is most important, creates a serious threat both for servicing personnel health and the environment. Currently one of the most serious alternatives of gaseous sterilization consists in the use of gas discharge plasma as sterilizing agent. The main advantage of plasma technique is given by the fact that plasma, as chemically active medium, is formed as a result of excitation, dissociation, and ionization

Numerical Simulation of Air Plasma Composition and Discussion

According to [4] and the experiments described below, in the sterilization by air barrier discharge, besides ozone, others active particles play role as well. In order to understand, what active particles are, and to find the plasma component content, we modeled barrier discharge in air under conditions close to experimental ones. Modeling of the plasma component content was performed separately for each of two parts of the working chamber – the discharge gap of barrier discharge and the sterilizing chamber itself, where processed items were placed.

In the calculation of the plasma component content and the concentrations of molecules and radicals formed immediately in barrier discharge, the following kinetic equations are used:

$$\frac{dN_i}{dt} = S_{ei} + \sum_j k_j N_j + \sum_{j,l} k_{jl} N_j N_l + \dots \quad (1)$$

Here, N_i are the concentrations of molecules and radicals; k_j, k_{jl} are the rate constants of molecular processes; S_{ei} is the rate of forming the products of electron-molecular reactions, which was calculated from the following equation:

$$S_{ei} = \frac{W}{V} \frac{1}{\epsilon_{ei}} \sum_j \frac{W_{ej}}{W_{ej} + W_j} \quad (2)$$

Here, W is the power introduced into barrier discharge; V is the discharge chamber volume; W_{ej} is the specific power spent to the electron-molecular process of nonelastic scattering with threshold energy ϵ_{ei} :

$$W_{ei} = \sqrt{\frac{2q}{m}} n_e N_i \epsilon_{ei} \int \epsilon Q_{ei}(\epsilon) f(\epsilon) d\epsilon, \quad (3)$$

where $q = 1.602 \cdot 10^{-12}$ erg/eV; m and n_e are the electron mass and concentration, respectively; Q_{ei} is the cross section of the respective nonelastic process; $f(\epsilon)$ is the electron energy distribution function (it was calculated from the Boltzmann equation [5]).

W_i is the specific power spent for the gas heating:

$$W_i = \frac{2m}{M_i} \sqrt{\frac{2q}{m}} n_e N_i \int_0^\infty \epsilon^2 Q_i(\epsilon) f(\epsilon) d\epsilon, \quad (4)$$

where M_i is the mass of a respective molecule; Q_i is the transport cross section of scattering.

The electron energy distribution function and specific powers W_{ei} and W_i are strongly dependent on the electric field in microscopic chaotically moving current channels in the discharge gap. Since the electric field in current channels changes in space and time in random way and in a rather wide range, the mean field value was used in the calculations. As is shown in [6], where barrier discharge in air at atmospheric pressure was studied, the mean electric field in current channels $E = 20$ kV/cm.

On solving Eqs. (1), more than 100 elementary processes were taken in consideration (see Table). (In the absence of the experimental data in the literature, the cross sections of nonelastic scattering of electrons by some molecules were calculated by the Thompson - Gryzinski formulas [13, 14].)

The calculation of the concentrations of atoms, molecules, and radicals in the sterilizing chamber was also performed on the basis of Eqs. (1), but with the substitution of S_{ei} by $S_i = \frac{N_i(\tau)}{\tau} \frac{V}{V_S}$, where τ is the time of the gas pumping through barrier discharge;

N	Reactions		Reference
1	2	3	4
1	$H_2O + e \rightarrow OH + H + e$	$1.66/n_e$	*
2	$O_2 + e \rightarrow O + O + e$	$7.5 \cdot 10^{-2}/n_e$	*
3	$O_2 + e \rightarrow O + O(d) + e$	$2.5 \cdot 10^{-2}/n_e$	*
4	$N_2 + e \rightarrow N + N + e$	$5.5 \cdot 10^{-3}/n_e$	*
5	$O(d) + H_2O \rightarrow OH + OH$	$2.2 \cdot 10^{-10}$	6
6	$O(d) + N_2 \rightarrow O + N_2$	$2.6 \cdot 10^{-11}$	6
7	$O(d) + O_2 \rightarrow O + O_2$	$3.8 \cdot 10^{-11}$	6
8	$O(d) + H_2O \rightarrow O + H_2O$	$1.2 \cdot 10^{-11}$	6
9	$N + HO_2 \rightarrow NO + OH$	$2.2 \cdot 10^{-11}$	6
10	$NO_3 + OH \rightarrow NO_2 + HO_2$	$2.3 \cdot 10^{-11}$	6
11	$OH + OH + M \rightarrow H_2O_2 + M$	$6.5 \cdot 10^{-31} (300/T)^{0.7}$	6
12	$OH + O_3 \rightarrow HO_2 + O_2$	$1.3 \cdot 10^{-12} e^{-956/T} + 2.3 \cdot 10^{-11} e^{110/T}$	6
13	$OH + HO_2 \rightarrow H_2O + O_2$	$4.8 \cdot 10^{-11} e^{250/T}$	6
14	$O + HO_2 \rightarrow OH + O_2$	$2.9 \cdot 10^{-11} e^{200/T}$	6
15	$H + O_3 \rightarrow OH + O_2$	$1.4 \cdot 10^{-10} e^{-480/T}$	6
16	$H + HO_2 \rightarrow OH + OH$	$3.0 \cdot 10^{-10} e^{-500/T}$	6
17	$H + OH + M \rightarrow H_2O + M$	$1.1 \cdot 10^{-23} T^{-2.6}$	6
18	$OH + OH \rightarrow H_2O + O$	$1.0 \cdot 10^{-11} e^{-500/T}$	6
19	$OH + H_2O_2 \rightarrow H_2O + HO_2$	$1.2 \cdot 10^{-11} e^{-270/T} + 8.0 \cdot 10^{-12} e^{2060/T}$	6
20	$O + O + M \rightarrow O_2 + M$	$2.76 \cdot 10^{-31/T}$	6
21	$HO_2 + HO_2 \rightarrow H_2O_2 + O_2$	$2.2 \cdot 10^{-13} e^{600/T}$	6
22	$HO_2 + HO_2 + M \rightarrow H_2O_2 + O_2 + M$	$1.9 \cdot 10^{-33} e^{980/T}$	6
23	$N + N + M \rightarrow N_2 + M$	$8.3 \cdot 10^{-34} e^{500/T}$	6
24	$O + H_2O_2 \rightarrow OH + HO_2$	$1.45 \cdot 10^{-15}$	7
25	$O + H_2O_2 \rightarrow H_2O + O_2$	$1.45 \cdot 10^{-15}$	7
26	$O(d) + O_3 \rightarrow O + O + O_2$	$2.33 \cdot 10^{-10}$	8
27	$O(d) + O_3 \rightarrow O_2 + O_2$	$2.33 \cdot 10^{-10}$	8
28	$H + HO_2 \rightarrow H_2O_2$	$9 \cdot 10^{-11}$	9
29	$N + O_3 \rightarrow NO + O_2$	$2 \cdot 10^{-16}$	10
30	$N + NO_2 \rightarrow N_2 + O_2$	$7 \cdot 10^{-13}$	10
31	$N + NO_2 \rightarrow N_2 + O + O$	$9.1 \cdot 10^{-13}$	10
32	$O + O_3 \rightarrow O_2 + O_2$	$2 \cdot 10^{-11} e^{2300/T}$	10
33	$O + NO_3 \rightarrow O_2 + NO_2$	$1 \cdot 10^{-11}$	10
34	$NO_3 + NO_3 \rightarrow O_2 + NO_2 + NO_2$	$2.3 \cdot 10^{-13} e^{-1600/T}$	10
35	$N + OH \rightarrow NO + H$	$3.8 \cdot 10^{-11} e^{85/T}$	11
36	$N + O_2 \rightarrow NO + O$	$4.4 \cdot 10^{-12} e^{-3220/T}$	7
37	$N + O + M \rightarrow NO + M$	$5.46 \cdot 10^{-33} e^{155/T}$	7
38	$NO + H + M \rightarrow HNO + M$	$3.4 \cdot 10^{-32}$	7
39	$NO + HO_2 \rightarrow NHO + O_2$	$9.1 \cdot 10^{-19} e^{2819/T}$	7
40	$NO + HO_2 + M \rightarrow HNO_3 + M$	$5.6 \cdot 10^{-33}$	7
41	$NO + HO_2 \rightarrow NO_2 + OH$	$3.7 \cdot 10^{-12} e^{240/T}$	7
42	$NO + N \rightarrow N_2 + O$	$3.1 \cdot 10^{-11}$	7
43	$NO + NO_3 \rightarrow NO_2 + NO_2$	$1.6 \cdot 10^{-11} e^{150/T}$	7
44	$NO + O + N_2 \rightarrow NO_2 + N_2$	$9.1 \cdot 10^{-28} T^{-1.6}$	7

1	2	3	4
45	$\text{NO} + \text{O}_3 \rightarrow \text{NO}_2 + \text{O}_2$	$2.0 \cdot 10^{-12} e^{-14400/T}$	7
46	$\text{NO} + \text{OH} + \text{M} \rightarrow \text{HNO}_2 + \text{M}$	$7.4 \cdot 10^{-31} (T/300)^{-2.4}$	7
47	$\text{NO}_2 + \text{HO}_2 + \text{M} \rightarrow \text{HO}_2\text{NO}_2$	$1.5 \cdot 10^{-31} (T/300)^{-3.2}$	7
48	$\text{NO}_2 + \text{N} \rightarrow \text{N}_2\text{O} + \text{O}$	$2.4 \cdot 10^{-12}$	7
49	$\text{NO}_2 + \text{N} \rightarrow \text{NO} + \text{NO}$	$6.0 \cdot 10^{-13}$	7
50	$\text{NO}_2 + \text{NO}_2 + \text{M} \rightarrow \text{N}_2\text{O}_4 + \text{M}$	$1.4 \cdot 10^{-33} (T/300)^{-3.8}$	7
51	$\text{NO}_2 + \text{NO}_3 + \text{M} \rightarrow \text{N}_2\text{O}_5 + \text{M}$	$2.7 \cdot 10^{-30} (T/300)^{-3.4}$	7
52	$\text{NO}_2 + \text{O} \rightarrow \text{NO} + \text{O}_2$	$6.5 \cdot 10^{-12} e^{120/T}$	7
53	$\text{NO}_2 + \text{O}_3 \rightarrow \text{NO} + \text{O}_2 + \text{O}_2$	$1.0 \cdot 10^{-18}$	7
54	$\text{NO}_2 + \text{O} + \text{M} \rightarrow \text{NO}_3 + \text{M}$	$9.0 \cdot 10^{-32} (T/300)^{-2.0}$	7
55	$\text{NO}_2 + \text{O}_3 \rightarrow \text{NO}_3 + \text{O}_2$	$1.2 \cdot 10^{-13} e^{-2450/T}$	7
56	$\text{NO}_2 + \text{OH} + \text{M} \rightarrow \text{HNO}_3 + \text{M}$	$2.2 \cdot 10^{-30} (T/300)^{-2.9}$	7
57	$\text{N}_2\text{O}_5 + \text{M} \rightarrow \text{NO}_3 + \text{NO}_2 + \text{M}$	$8.8 \cdot 10^{-6} e^{-9700/T}$	7
58	$\text{N}_2\text{O}_5 + \text{H}_2\text{O} \rightarrow \text{HNO}_3 + \text{HNO}_3$	$5.0 \cdot 10^{-21}$	7
59	$\text{HNO} + \text{O}_2 \rightarrow \text{NO} + \text{HO}_2$	$5.25 \cdot 10^{-12} e^{-1510/T}$	7
60	$\text{HNO}_2 + \text{OH} \rightarrow \text{NO}_2 + \text{H}_2\text{O}$	$1.8 \cdot 10^{-11} e^{-39/T}$	7
61	$\text{HNO}_3 + \text{OH} \rightarrow \text{NO}_3 + \text{H}_2\text{O}$	$1.5 \cdot 10^{-14} e^{650/T}$	7
62	$\text{HNO}_3 + \text{NO} \rightarrow \text{HNO}_2 + \text{NO}$	$7.37 \cdot 10^{-21}$	7
63	$\text{HO}_2\text{NO}_2 + \text{M} \rightarrow \text{HO}_2 + \text{NO}_2 + \text{M}$	$5.0 \cdot 10^{-6} e^{-10000/T}$	7
64	$\text{O} + \text{O}_2 + \text{M} \rightarrow \text{O}_3 + \text{M}$	$6.2 \cdot 10^{-34} (T/300)^{-2.0}$	7
65	$\text{O} + \text{OH} \rightarrow \text{O}_2 + \text{H}$	$2.3 \cdot 10^{-11} e^{110/T}$	7
66	$\text{H} + \text{O}_2 + \text{M} \rightarrow \text{HO}_2 + \text{M}$	$4.8 \cdot 10^{-32}$	7
67	$\text{H} + \text{OH} + \text{M} \rightarrow \text{H}_2\text{O} + \text{M}$	$8.6 \cdot 10^{-31}$	7
68	$\text{O}_3 + e \rightarrow \text{O}_2 + \text{O} + e$	$7.3 \cdot 10^{-3} / n_e$	*
69	$\text{N}_2\text{O} + e \rightarrow \text{N}_2 + \text{O} + e$	$1.68 \cdot 10^{-2} / n_e$	*
70	$\text{NO}_2 + e \rightarrow \text{NO} + \text{O} + e$	$7.5 \cdot 10^{-2} / n_e$	*
71	$\text{N}_2\text{O}_4 + e \rightarrow \text{NO}_2 + \text{NO}_2 + e$	$2.5 \cdot 10^{-2} / n_e$	*
72	$\text{N}_2\text{O}_5 + e \rightarrow \text{NO}_2 + \text{NO}_3 + e$	$2.5 \cdot 10^{-2} / n_e$	*

* rates were calculated with the use of the electron energy distribution function.

$N_i(\tau)$ is the concentration of mixture components in the discharge at the time τ , and V_S is the sterilizing chamber volume. Electron and ion processes in the sterilizing chamber were not taken into account due to the absence of charged plasma particles in it.

Eqs. (1) were solved together with the Boltzmann equation by means of numerical methods analogous to those in [5].

In Figs. 1, 2, the component contents of a mixture in the sterilizing chamber in dry air (water vapor is absent) of various times τ of pumping through barrier discharge ($\tau = \frac{V}{Sv}$, where S is the cross section of the discharge chamber, v is the pumping rate) are given. The first figure corresponds to $\tau = 0.1$ s; the second one - to a higher pumping rate, when it is possible to neglect the destruction of the products of plasma-

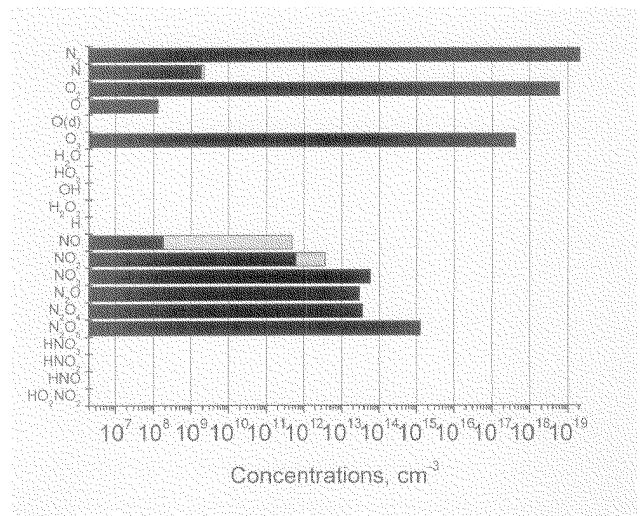


Fig. 1. Component content of particles in the sterilizing chamber under barrier discharge operation in dry air (0% RH) for the pumping time through the discharge gap $\tau = 0.1$ s (gas flow is 1 l/min)

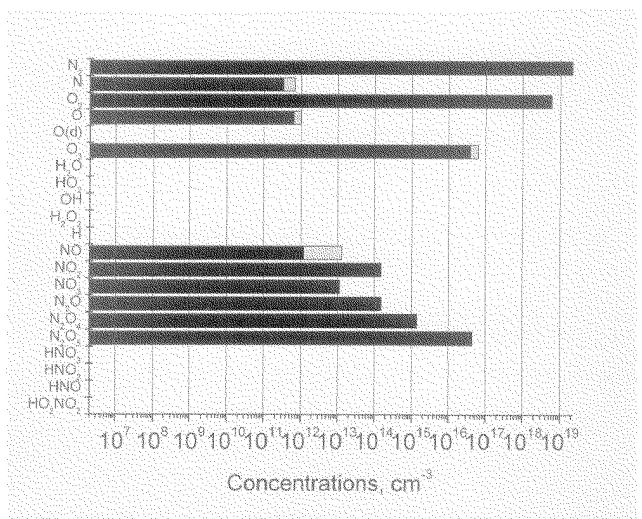


Fig. 2. Component content of particles in the sterilizing chamber under barrier discharge operation in dry air (0% RH) for the pumping time through the discharge gap $\tau = 0$

chemical reactions in the discharge chamber by electrons. Dark strips correspond to the concentrations of mixture components after 30 minutes of the discharge operation; light strips represent maximum achievable values of the densities during the mentioned time period. The largest values are reached by O_3 and N_2O_5 concentrations. As one can see from the figures, the densities of particular mixture components in the sterilizing chamber possess a rather strong dependence on the pumping rate. In this case, the concentrations of certain components increase, whereas the concentrations of other ones decrease with variation of the pumping rate. With a decrease of the pumping

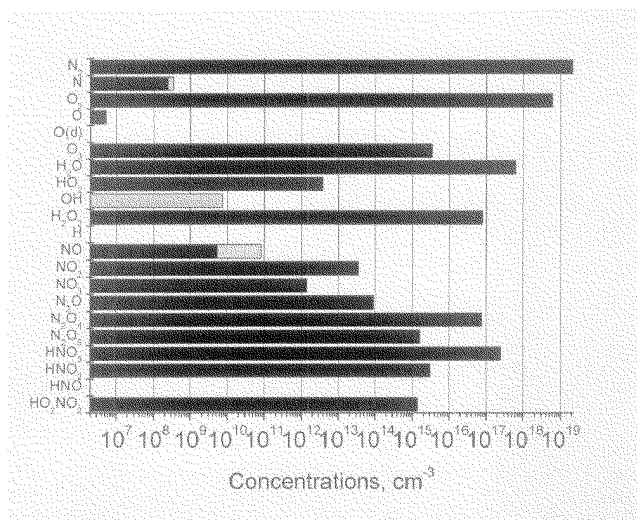


Fig. 3. Component content of particles in the sterilizing chamber under barrier discharge operation in moist air (100% RH) for the pumping time through the discharge gap $\tau = 0.1$ s (gas flow is 1 l/min)

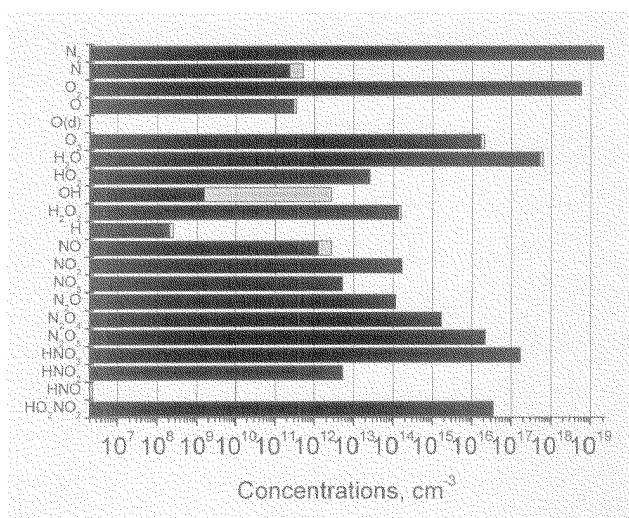


Fig. 4. Component content of particles in the sterilizing chamber under barrier discharge operation in moist air (100% RH) for the pumping time through the discharge gap $\tau = 0$

rate, the amount of nitrogen-containing molecules decreases, however, the ozone density grows up. It is due to the fact that, at a low pumping rate of the mixture through the discharge gap, the essential decomposition of nitrogen-containing molecules occurs due to the dissociation by electron hits. And, in turn, it results in a growth of the ozone concentration via the sequence of chain reactions.

In Figs. 3, 4, component contents are presented for the mixture in the sterilizing chamber in case of moist air (H_2O concentration corresponds to the partial pressure of saturated water vapor) for two given above rates of pumping through the discharge chamber.

It is clearly seen from the comparison of Figs. 3 and 4 with Figs. 1 and 2 that moistening the air results in the following effects:

1. Ozone concentration decreases significantly, and its dependence on the pumping rate changes to the opposite one - an increase of the pumping rate results in an increase of the O_3 concentration.

2. N_2O_2 concentration increases by a factor of several orders of magnitude at a low pumping rate, and it exhibits a small variation at a high pumping rate.

3. Efficient generation of H_2O_2 , HNO_3 , HNO_2 and HO_2NO_2 molecules occurs, and their concentrations can essentially exceed the ozone concentration.

Experimental Set-up and Results

The experimental set-up was described in detail in [4]. It consists of a glass work chamber with a volume of 7 liters, an electrode unit, holders for samples to be sterilized, and gas and electric power supply systems. The electrode unit was placed at the center of the chamber. Gases (air, O_2 , N_2 , Ar) with different humidity (RH) were supplied into the electrode unit, and chemically active particles generated by discharges were directed to the samples. A gas flow was varied in the range 0.5 - 10.0 l/min. Barrier discharge was created with pulsed power supply. The pulse duration was about 10 μ sec, and the pulse rates were up to 10 kHz. Specific power normalized to the volume of the discharge gap of barrier discharge $W_d = 2 \div 4$ W/cm³. The whole working chamber volume was spatially split into two parts - the central one, where the plasma generation occurred, and the sterilizing one, where processed articles were placed. The ratio of the volume of the discharge gap to that of sterilizing chamber comprised about $3 \cdot 10^{-3}$. The holders surrounded the electrode unit, and the mean distance from the discharge zone to the samples was about 10 cm.

Polished stainless steel strips inoculated with 10^6 spores *Bac. stearotherophilus* ATCC 7953 (the surface spore density $\geq 0.5 \cdot 10^6$ sp/cm²) were used as bio-indicators (BI). After the processing, the samples were treated by the standard method used in microbiology, and then the number of surviving bacteria was determined by using the pour-plate method.

The data for sterilization by barrier discharge are given in Figs. 5, 6. As one can see from Fig. 5, the least efficiency of the sterilization is in the cases of nitrogen and argon. In the case of dry gases, the most efficient processing is observed in oxygen (due to the highest ozone concentration). Oxygen moistening results in a decrease of the ozone concentration, however, the efficiency of the sterilization practically is

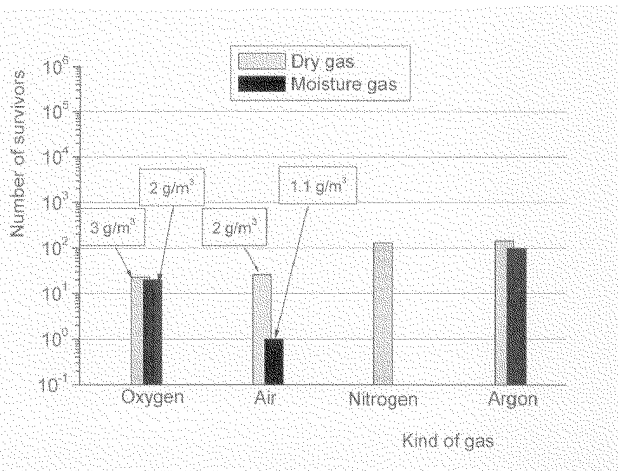


Fig. 5. Comparison of the sterilizing ability of barrier discharge in various dry (45% RH) and moist (90% RH) gases by suppression of the number of survived spores *Bac. stearothermophilus* at metal strips. Initial bacterial loading is $8 \cdot 10^5$ spores. Sterilization time is 60 min; gas flow is 1 l/min, $W_d = 2$ W/cc; ozone concentration is shown for oxygen and air

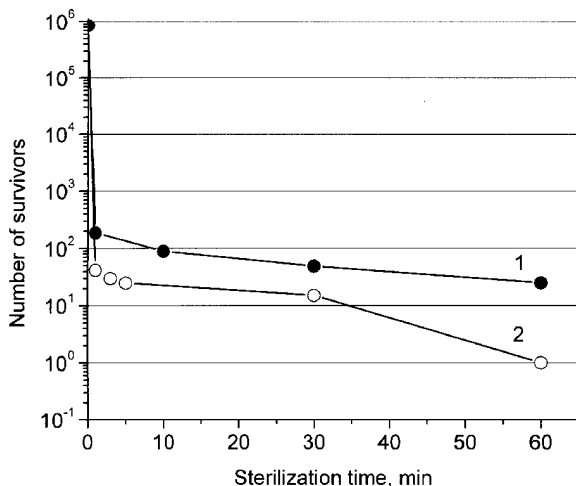


Fig. 6. Survival curves for spores *Bac. stearothermophilus* at metal strips obtained by the colony count method under sterilization by barrier discharge in dry (45% RH) (curve 1) and moist (90% RH) (curve 2) air. Initial bacterial loading is $8 \cdot 10^5$ spores. Gas flow is 1 min^{-1} , $W_d = 2 \text{ W/cm}^{-3}$

not changed at that. This fact given an evidence that the sterilizing action of ozone is enhanced in the presence of moisture in the chamber. At the same time, moistening the air (which is also accompanied by a decrease of the ozone concentration) results in the essential diminishing of the number of survived spores. As will be shown below, this effect is most likely due to the fact that, in addition to ozone, other chemically active particles participating in the sterilization are also created - first of all, they are hydrogen peroxide, nitrogen oxides, and nitric and nitrous acids.

Fig. 6 exhibits the survival curves (that is, the dependences of the number of survived spores on the processing time) obtained at sterilization with the use of barrier discharge in dry and moist air. One can see from the figure that, during initial several minutes, the number of survived spores decreases significantly with time (practically with the same 'speed' for dry and moist air), and in subsequent the decrease of the number of survived spores occurs much more slowly. Difference in biological activity of the active media generated by barrier discharge in dry and wet air is exhibited especially at the second stage - the number of survived spores in case of wet air is essentially lower.

Since experimental conditions correspond to those given in Figs. 1 and 3, it follows from their comparison that the observed increase of the sterilization efficiency under air moistening is due to the generation of biologically active particles, hydrogen peroxide H_2O_2 , nitric HNO_3 and nitrous HNO_2 acids, and HO_2NO_2 radicals, and to the growth of N_2O and N_2O_5 concentrations.

Thus, as follows from theoretical calculations and the experiment, by means of variations of the pumping rate, one can control the concentrations of biologically active particles in a wide range and, consequently, optimize the process of sterilization of medical instruments.

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