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STUDY OF THE NITROCELLULOSE GUNPOWDER REGENERATION PROCESS USING HYDROGEN PEROXIDE

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Abstract

World experience in storing ammunition shows that during long-term storage, pyroxylin gunpowders used in ammunition are capable of arbitrarily undergoing various physical and chemical transformations, which negatively affects the ballistic characteristics of ammunition. In some countries, ammunition is in use with a shelf life of 30–35 years or more.

Factors influencing the physicochemical stability of gunpowder charges during long-term storage have been identified.

An analysis of the chemical processes occurring in nitrocellulose gunpowders (NCP) during long-term storage is carried out, and the possibility of regenerating NCP by treatment with hydrogen peroxide is substantiated.

It is indicated that there are no methods for regenerating gunpowder charges with long shelf life. It has been shown that one of the ways to increase the ballistic and energy characteristics of long-term storage ammunition can be the regeneration of nitrocellulose gunpowder charges.

Data are presented on an experimental study to determine the characteristics of NCP after regeneration. It has been shown that when regenerating nitrocellulose gunpowder charges with hydrogen peroxide, the calorific value of the starting material increases.

Based on the results obtained, an approach to assessing the feasibility of using regeneration technology is proposed. This approach is based on the effect of mass replenishment during the regeneration process due to ongoing chemical processes and heat and mass transfer.

The ways for further research on the use of nitrocellulose as a high-calorie fuel are presented.

Keywords: nitrocellulose, pyroxylin gunpowder, homologation, gunpowder regeneration, hydrogen peroxide, performance prediction.

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1. Introduction

The ballistic qualities of pyroxylin gunpowders are determined by a combination of a number of properties: performance, shape of the gunpowder elements, combustion rate [1–3]. These qualities are assessed by the values of the initial velocity of the projectile, the maximum pressure of the gunpowder gases [3–5] and the median deviation of the initial velocity when firing from a particular weapon [1, 4, 6].

Nitrocellulose gunpowders (NCPs) are unstable chemical compounds. This is due to the fact that the ballistic stability of NCP is limited due to the autocatalytic reaction, the rate of which can vary significantly under the influence of temperature and moisture. A change in the content of gunpowder components is accompanied by a significant change in its ballistic properties. When the content of volatile substances in gunpowder changes by 1 %, the combustion rate changes by 10–12 %, the pressure of gunpowder gases by 9–16 %, and the initial velocity of the projectile by 3–4 % [2, 7].

Ballistic stability plays a decisive role in determining the service life of propellants [1, 2, 8–10]. The duration of storage of gunpowder is of exceptionally great importance, since it is related to the issues of the required production capacity of gunpowder factories and the system for monitoring the condition of gunpowder. However, establishing guarantee periods for the safe storage of gunpowders and their operational suitability is a complex problem.

Low-resistant gunpowder is not only economically unprofitable, since frequent periodic renewal of the combat stock is necessary, but also poses a significant danger. The problem of storing and accumulating large quantities of ammunition, namely gunpowder charges, is typical not only for Ukraine, but also for developed NATO countries. There are known cases when spontaneous combustion of gunpowder was accompanied by human casualties, and sometimes took on the dimensions of major disasters [1, 9]. For example, an explosion in the cannon turret of the battleship Iowa (USA, 1989), ruptures of artillery system barrels (Germany), an explosion at an ammunition depot (France, 2007), explosions of ammunition depots in Vrbetica (Czech Republic, 2014).

The problem of aging gunpowder charges and, in connection with this, deterioration of the ballistic characteristics of weapons is known and not new. There are data from studies of physicochemical changes in gunpowder, covering a shelf life of up to 5 years, forecasts – up to 10 years [1, 2, 9]. In works [11–13], data were obtained on the changes occurring in gunpowder during longer storage periods (17–21 years).

There are warranty periods for storing ammunition, which are determined by the following conditions:

– shelf life up to 10 years;

– during the storage period, the initial velocity of the projectile should not decrease by more than 5 % of the Table value.

Therefore, by long shelf life let's mean such a period when the shelf life of 10 years is exceeded and (or) the initial speed has decreased by more than 5 %.

The problem of stabilizing gunpowder charges arises during the development and adoption of them for service. The relevance of this task arises both from the requirements for ensuring safety in the handling of gunpowder, and from the requirements of an economic nature and ensuring the reliability of the functioning of ammunition charges.

This gives rise to a complex problem related to:

– possibility of using these ammunition;

– restoration of their properties (homologation);

– definition of limiting terms when homologation is still possible;

– disposal (destruction).

Many researchers have dealt with this topical problem [1–3, 8–16]. Despite the large volume of work performed, there are still many unresolved issues related to the complexity of the physical and chemical processes occurring in gunpowder during storage. A certain difficulty is caused by monitoring the progress of these processes in multicomponent rigid systems (especially at intermediate stages), and the impossibility of isolating most of the transformation products in pure form. Theoretically, it also has no development due to the lack of a valid chemical formula for NCP.

A large amount of experimental material has been accumulated on the reactivity of aromatic compounds, and the basic principles of electronic theory have been developed, establishing a connection between the chemical structure of substances and their reactivity. However, these data should already be considered outdated, since they were obtained for service periods of 19–26 years, and therefore can only be used to generalize the patterns of changes in the NCP physicochemical properties.

Available sources [12, 16–21] devoted to the operation, study of the properties and life cycles of NCPs, contain information about the influence of modified characteristics of gunpowders on the material part and ballistic characteristics of weapons. However, in [12, 16–21] the issues of regeneration and restoration of the properties of gunpowders are not considered.

Obviously, this is due to the fact that, through the planned rotation of ammunition at certain time periods, as a rule, long-term storage of ammunition was not allowed. However, the armed forces of developed countries (USA, Germany, UK, France) have quite large stocks of ammunition [22–24], which require disposal or regeneration, since their storage periods are very long.

Thus, when analyzing available open domestic and foreign sources [4–10, 12, 16–21], no data was found on the results of treating nitrocellulose compounds, in particular gunpowders, with hydrogen peroxide.

In connection with the above, the aim of the study is to develop a method for regenerating nitrocellulose gunpowder charges with long shelf life, allowing to restore their properties for the further use of ammunition for its intended purpose.

To achieve this aim, it is necessary to solve the following objectives:

1. Develop a method for regenerating nitrocellulose gunpowder charges with long shelf life.

2. Conduct experimental studies to determine the characteristics of nitrocellulose gunpowders after regeneration.

3. Assess the effect of gunpowder charge regeneration and the possibility of increasing the service life of ammunition.

2. Materials and methods of research

The object of the experimental study is the process of changing the ballistic characteristics of nitrocellulose gunpowder charges when exposed to hydrogen peroxide.

During the study, the main hypothesis was put forward, which is that when gunpowder elements are treated with hydrogen peroxide, the strength of the gunpowder increases, which is one of the main ballistic characteristics of the charge. The strength of gunpowder depends on its mass and calorific value. This research hypothesis was tested experimentally by conducting a series of physical experiments.

Thus, the study consisted of assessing the ability of fuel elements to regenerate properties during exposure to hydrogen peroxide. The experimental research program for treating a gunpowder charge with a solution of hydrogen peroxide is given in [17], and the author's experimental technique is described in [20].

The experimental study consisted of 10 replicate runs for two types of gunpowder charges. Each series used 10 elements of a sample of these types of gunpowder.

In accordance with the methodology [20], each series of experiments consisted of 4 stages:

I – determination of the mass of gunpowder elements;

II – treatment of gunpowder elements with a solution of hydrogen peroxide and drying them in a laboratory oven at a temperature of 60 °C;

III – determination of the mass of gunpowder elements after drying;

IV – combustion of control (untreated) samples and samples treated with hydrogen peroxide.

The study used a 4Zh40 tank shot gunpowder charge. Its structure is shown in **Fig. 1**.

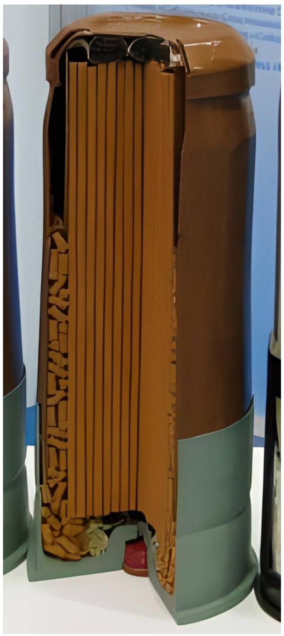

Fig. 1. Tank shot charge 4Zh40

Samples of 4Zh40 tank shot charge gunpowders are shown in **Fig. 2**.

a *b*

Fig. 2. Samples of 4Zh40 tank shot charge gunpowders after long-term storage:
a – seven-channel; *b* – single-channel

The experimental study used elements of seven-channel (7c) (**Fig. 2, *a***) and tubular single-channel (1c) (**Fig. 2, *b***) gunpowder manufactured in 1983 and a 47 % hydrogen peroxide solution.

The mass was measured on chemical laboratory scales, metrologically verified, with a measurement error of 0.001 g. The combustion rate of the gunpowder grains was measured using a stopwatch. Based on the purpose of the experiment, it was not the absolute value of the combustion rate that was considered more important, but its change after processing the gunpowder.

Processing of experimental data was carried out according to the well-known method of statistical processing of empirical data [25] and included checking for the presence of outlier observations, checking the reproducibility of experiments, and checking the adequacy of the model.

The main simplifications and assumptions used when processing experimental data:

1. The conditional theoretical ideal formula of nitrocellulose was used and the chemical composition of gunpowder after its long-term storage was not determined accurately enough.

2. The increase in the mass of gunpowder after treatment with hydrogen peroxide occurs only due to hydrogen.

3. It is known that hydrogen peroxide whitens legnin and if there is a free bond in the structure ($C_6H_7O_2(OH)_3$), then hydrogen can replace it. However, the interaction of hydrogen peroxide with carbon contained in nitrocellulose in the form of lignin is not considered due to the lack of data on such reactions.

4. The combustion of gunpowder does not occur in a closed volume.

3. Results and discussion

3. 1. Method for regenerating gunpowder charges based on nitrocellulose

Based on the hypothesis put forward, a method is proposed for the regeneration of nitrocellulose-based gunpowder charges, which consists of the action of hydrogen peroxide on the elements of gunpowder, which allows for partial homologation of the ballistic and energy characteristics of gunpowder charges.

Its essence is as follows.

During the fuel production process, the nitriding reaction is the most typical and consists of replacing hydrogen atoms in organic compounds with the nitro group NO_2. Therefore, cellulose nitriding occurs as follows [26, 27]:

$$C_{24}H_{28}O_8\left(OH\right)_{12} + 8HNO_3 = C_{24}H_{28}O_8\left(ONO_2\right)_8\left(OH\right)_4 + 8H_2O,$$

$$C_{24}H_{28}O_8\left(ONO_2\right)_8\left(OH\right)_4 + HNO_3 = C_{24}H_{28}O_8\left(ONO_2\right)_9\left(OH\right)_3 + H_2O, \qquad (1)$$

$$C_{24}H_{28}O_8\left(ONO_2\right)_{11}OH + HNO_3 = C_{24}H_{28}O_8\left(ONO_2\right)_{12} + H_2O,$$

where $C_{24}H_{28}O_8\left(OH\right)_{12}$ – the conditional formula of the original cellulose.

As is known [26, 27], complete nitration of cellulose stops when the nitrogen concentration reaches 14.14 % in products of the $C_{24}H_{28}O_8(ONO_2)_{12}$ type. For this reason, the products of this reaction contain compounds with incomplete nitration. The level of formation of incomplete nitration depends on the level assessed in the final product by the percentage of nitrogen (pyroxylin or colloxylin).

Therefore, the so-called general formula of nitrocellulose is used [27]:

$$C_6H_7O_2\left(OH\right)_{3-x}\left(ONO_2\right)_x|_4, \qquad (2)$$

where $x = 1, 2, 3$ – number of ONO_2 groups. Empirical formulas are used to describe the conventional value of cellulose nitrates, which consist of a certain amount of nitrogen in 1 kg of fuel.

Thus, pyroxylin containing 13 % nitrogen is represented by the formula [26]:

$$C_{21.551}H_{25.638}O_{36.520}N_{9.280}. \qquad (3)$$

Thus, the formula of nitrocellulose depends on the nitrogen content, which is why the generalized chemical formula (2) is used.

3. 2. Results of experimental studies to determine the characteristics of nitrocellulose gunpowders after regeneration

To test the effectiveness of the above method for regenerating a nitrocellulose-based gunpowder charge, experimental studies were carried out.

The experimental results made it possible to estimate the change in the mass and combustion rate of gunpowder elements before and after their treatment with hydrogen peroxide. **Table 1** presents the data of one series of experiments for two types of NCP elements.

Table 1
Results of an experiment on the effects of hydrogen peroxide on types of gunpowder charge elements

Gunpowder		Mass, g			Combustion speed, mm/s		
Type	**No.**	**Before processing**	**After processing**	**Change**	**Before processing**	**After processing**	**Change**
7c	1	0.67	0.675	+0.005	5.8	6.2	+0.4
	2	0.669	0.674	+0.005	5.78	6.19	+0.41
	3	0.671	0.677	+0.006	5.79	6.22	+0.43
	4	0.67	0.675	+0.005	5.81	6.19	+0.38
	5	0.671	0.675	+0.004	5.81	6.21	+0.4
	6	0.669	0.675	+0.006	5.79	6.2	+0.41
	7	0.669	0.674	+0.005	5.82	6.21	+0.39
	8	0.67	0.675	+0.005	5.81	6.21	+0.4
	9	0.671	0.675	+0.004	5.79	6.18	+0.39
	10	0.67	0.675	+0.005	5.8	6.19	+0.39
Series average		0.67	0.675	+0.005	5.8	6.2	+0.4
1c	1	1.01	1.024	+0.014	6.27	6.94	+0.67
	2	1.011	1.026	+0.015	6.29	6.98	+0.69
	3	1.01	1.025	+0.015	6.28	6.97	+0.69
	4	1.01	1.025	+0.015	6.27	6.95	+0.68
	5	1.011	1.024	+0.013	6.22	6.86	+0.64
	6	1.009	1.026	+0.017	6.4	7.1	+0.7
	7	1.01	1.024	+0.014	6.27	6.93	+0.66
	8	1.009	1.026	+0.017	6.39	7.11	+0.72
	9	1.01	1.025	+0.015	6.26	6.92	+0.66
	10	1.01	1.025	+0.015	6.27	6.95	+0.68
Series average		1.01	1.025	+0.015	6.29	6.97	+0.68

After processing the results of all series of the experiment, averaged controlled data were obtained before and after exposure to hydrogen peroxide. **Table 2** shows the averaged experimental data and characteristic features.

Table 2
Averaged experimental data

Type	Mass, g			Combustion speed, mm/s			Colour	
	Before processing	**After processing**	**Change**	**Before processing**	**After processing**	**Change**	**Before processing**	**After processing**
7c	0.670	0.675	+0.005	5.8	6.2	+0.4	dark at the ends	everything is dark
1c	1.01	1.025	+0.015	6.29	6.97	+0.68	light	without change

Effects observed during the experiment:

– gas bubbles were observed all the time around the surface of the elements, varying in size by 4–5 times. Some areas were covered in small bubbles;

– at the end of the experiment (after 2 hours), hydrogen peroxide had a light-yellow color;

– the ends of the elements of gunpowder 7c acquired a dark color after exposure;

– the mass of gunpowder elements 1c and 7c has been increased;

– the combustion rate of the gunpowder elements is slightly higher than the control samples;

– the flame area of the gunpowder elements 1c is wider than that of the control samples (**Fig. 3**), which indicates an increase in energy value.

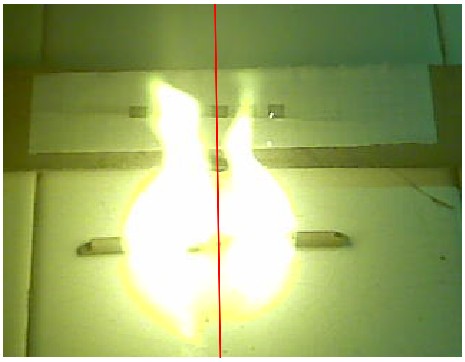

Fig. 3. The combustion process of gunpowder elements 1c:
a – sample after exposure to hydrogen peroxide; *b* – control sample

The effect of uneven combustion of gunpowder charge elements during a shot, described in [4], was also observed during experimental studies.

During the described experiment, the colors of the gunpowder elements were assessed visually by photographing the gunpowder elements before and after exposure to hydrogen peroxide [16].

When determining the mass, the measurement error was 0.001 g. It follows from this that the data in **Table 1** in terms of mass change exceed the measurement error and, thus, are a reliable result.

It has been experimentally shown that as a result of regeneration, the mass of nitrocellulose increases by the amount of adsorbed elements during its treatment with hydrogen peroxide, namely by 0.005 and 0.015 grams (**Table 2**). Thus, the increase in the mass of elements of seven-channel (7c) and tubular single-channel (1c) gunpowder, which are treated with hydrogen peroxide, is approximately 0.75 and 1.5 %, respectively.

Considering that the calorific value of hydrogen is characterized by a value of 120.9 MJ/kg, and the calorific value of nitrocellulose gunpowder is 5.9 MJ/kg, to equivalently compensate for the lost mass, gunpowder requires 20 times less mass of hydrogen. In an experimental study, as shown in **Table 2**, an increase in mass of up to 1.5 % was observed due to the presence of hydrogen, which leads to an increase in calorific value.

Analysis of the internal ballistic correction formulas shows that the increase in the mass of the gunpowder charge element is directly proportional to the change in speed. For this reason, one can expect a corresponding increase in the initial velocity when using a gunpowder charge treated with hydrogen peroxide within up to 5 % deviations from the table values (at the stage of preservation of propellant charges for 27–28 years).

It is useful to emphasize in this regard the importance of the task of restoring the properties of the gunpowder charge (full or partial) for a possible increase in shelf life and further use for its intended purpose. This becomes especially important when considering the volume of ammunition stored, the service life of which is steadily increasing every day.

Based on the results of experimental studies and the theoretical (ideal) formula of nitrocellulose, the fundamental possibility of a reaction on its surface with hydrogen peroxide is shown. The obtained experimental effect requires further research in the direction of considering nitrocellulose as an energy fuel.

Currently, there is a problem in the world with the use of hydrogen as fuel. Therefore, further research on the use of nitrocellulose as a hydrogen adsorbent is of interest. Both nitrocellulose and hydrogen are flammable, fuel substances. If it is possible to saturate nitrocellulose with hydrogen, it is possible to obtain hydrogen high-calorie fuel, which, due to its predicted properties, can be used both in mobile power plants and in stationary ones.

3. 3. Evaluation of the regeneration effect of the gunpowder charge

Based on the results obtained, an approach to assessing the feasibility of using regeneration technology is proposed. This approach is based on the effect of mass replenishment during the regeneration process due to ongoing chemical processes and heat and mass transfer. For ammunition of artillery systems in long-term storage, a reduction in charge mass has been established [13]. As a result of the regeneration process, the mass of gunpowder increases slightly, and thus part of the mass lost during storage is compensated. By tracing the relationship between the mass of the charge and the initial velocity of the projectile, it is possible to evaluate the effect of regeneration of the gunpowder charge.

At a fundamental level, in [27] the correlation of these parameters is presented, obtained as a result of the analysis of a significant number of results of experimental studies of various ammunition (ranging from unitary to separate-case loading for artillery systems).

From the correction formulas of internal ballistics [7, 13, 21] it is known that:

$$\frac{\Delta V_0}{V_0^{tab}} = k\frac{\Delta m}{m^{tab}}, \tag{4}$$

where ΔV_0 – change in initial speed; V_0^{tab} – table value of the initial speed; k – coefficient that can be calculated based on the results of firing with ammunition of a specific type; Δm – change in charge mass; m^{tab} – table value of the charge mass.

Expression (4) shows that a decrease in the mass of gunpowder leads to a decrease in the initial velocity of the projectile [13, 21].

Moving from finite increments ΔV_0, Δm to indivisible ones, let's obtain:

$$\frac{dV_0}{V_0^{tab}} = k\frac{dm}{m^{tab}}, \tag{5}$$

where V_0 – the initial speed; m – charge mass.

If for a certain period of time the gunpowder charge is stored $k = \text{const}$, then:

$$A = k\frac{dm}{m^{tab}} = \text{const}, \tag{6}$$

where A – a function depending on the caliber, strength of gunpowder, type of system (rifled barrel, smooth-bore).

The physical meaning of this function is that it shows what part of the initial velocity is accounted for by the restored charge mass.

Then, up to a constant coefficient C, the velocity function $V(m)$ is described by the linear dependence:

$$V(m) = A \cdot m + C. \tag{7}$$

Score C provided when, $m = 0$; $V_0 = 0$ leads us to the conclusion that $C = 0$.

Thus, let's finally obtain $V(m) = A{\cdot}m$, $\tan(\alpha) = A$ (**Fig. 4**).

According to research data, it is possible to predict an additional resource of the 4Zh40 $\Delta\tau_+$ gunpowder charge, during which the initial speed will not be less than the limit of 0.95 V_0 after exposure to a hydrogen peroxide solution (**Fig. 5**).

According to the forecast, the additional resource of the 4Zh40 gunpowder charge for a tank gun can be $\Delta\tau_+ = 9$–12 years under the same conditions and restrictions. This creates additional time for the production or purchase of new gunpowders in the absence of ammunition production means. In addition, taking into account the presence of arsenals with ammunition with long shelf life in many countries of the world, the resulting effect, brought to the level of industrial technology, will allow, if not eliminate, then significantly reduce the volume of disposal of this kind of gunpowder charges and thereby significantly save money on ordering new ones.

The article describes a physicochemical experiment confirming the regeneration effect exclusively in laboratory conditions. Therefore, with the industrial application of this technique, additional research is necessary that will make it possible to quantify the regeneration effect and select the optimal conditions for regeneration for each batch of ammunition, depending on the storage period (more than 15 years).

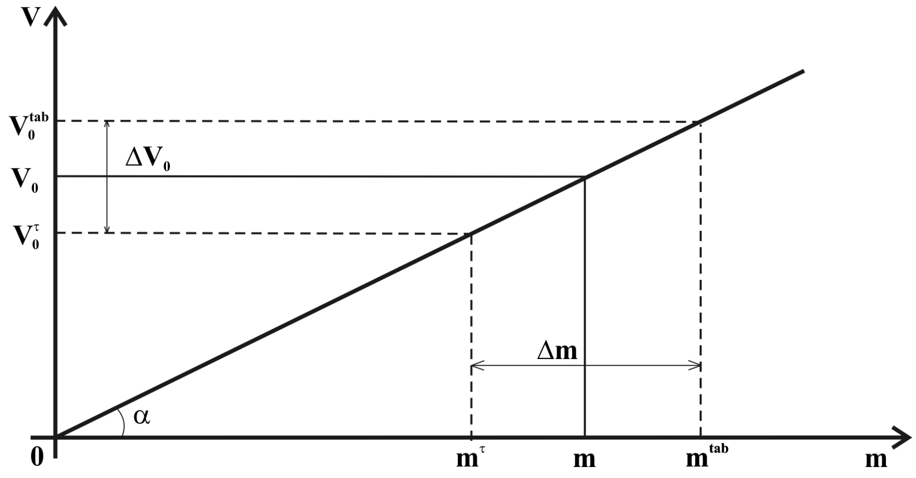

Fig. 4. Dependence of the change in the initial velocity of the projectile on the change in the charge mass (m^{τ} – mass of the charge at an arbitrary moment of time τ in the life cycle of the gunpowder charge, α – the angle of inclination)

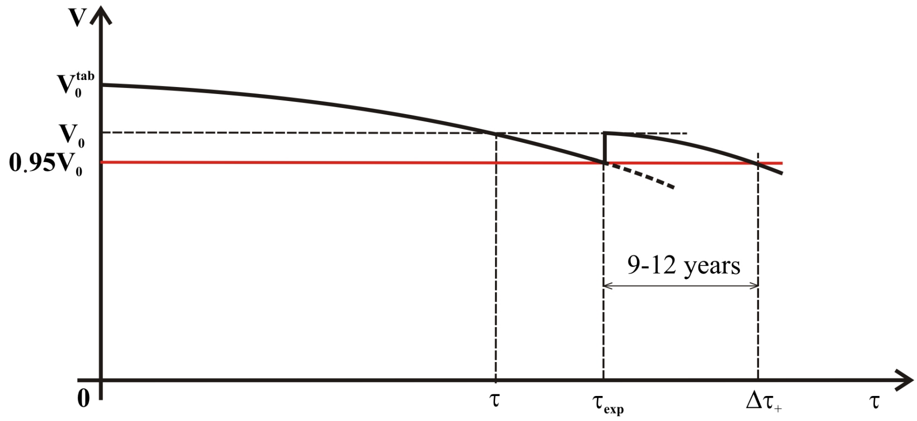

Fig. 5. Estimation of additional service life (τ_{exp} – regeneration time of the gunpowder charge)

The limits of applicability of this technique for any period of storage of ammunition are assessed by the possibility of restoring the ballistic characteristics of gunpowder to a value of more than 0.95 % of the table values.

Of particular interest are studies on the use of nitrocellulose (or lignin) as a hydrogen adsorbent to produce high-calorie fuel for transport and energy.

4. Conclusions

A method has been proposed for the regeneration of nitrocellulose gunpowders, which consists in the action of a 47 % hydrogen peroxide solution on the elements of gunpowder, which allows for partial homologation of the ballistic and energy characteristics of gunpowder charges of long-term storage ammunition.

Based on the results of experimental studies and the theoretical (ideal) formula of nitrocellulose, the fundamental possibility of a reaction on its surface with hydrogen peroxide is shown.

It has been experimentally shown that as a result of regeneration, the mass of nitrocellulose increases by the amount of adsorbed elements during its treatment with hydrogen peroxide. In this regard, the combustion rate of gunpowder increases and its calorific value as fuel increases.

In terms of calorific value, hydrogen is characterized by a value of 120.9 MJ/kg, while pyroxylin gunpowder at a constant volume is 5.9 MJ/kg. Thus, to equivalently compensate for the lost mass of gunpowder, a 20 times smaller mass of hydrogen is required.

Predicting changes in the characteristics of a gunpowder charge is one of the tasks associated with the problem of operating ammunition. At different storage times, the most important indicator of changes in the properties of a gunpowder charge is the initial velocity of the projectile.

According to the results of the forecast based on the data on the regeneration, the gunpowder mass will increase, which, in turn, leads to an increase in the initial speed by up to 5 %. This result will make it possible to increase the service life of ammunition by an additional 9–12 years.

The obtained experimental effect requires further research in the direction of considering nitrocellulose as an energy fuel.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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Data availability

Manuscript has no associated data.

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