Research and Development of Solid Fuel Compositions for the Production of Hydrogen

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 Received September 10, 2019; revised October 12, 2019; accepted November 14, 2019

Abstract—The pyrotechnic base of solid fuels of different compositions is investigated in order to produce hydrogen in the mode of the forced convection of combustion products. Potassium borohydride is used as the fuel, and metal oxides and hydroxides are used as the oxidizing agents. In each case, the formula for the fuel with the optimal percentage of components is developed. The studies have shown that among the metal oxides used as oxidizing agents (CuO, Fe₂O₃, MnO₂, and MoO₃), MnO₂ has the highest gas productivity. When metal hydroxides Ni(OH)₃, Al(OH)₃, and H₃BO₃ are used as oxidizing agents, Ni(OH)₃ can provide the maximum gas production.

Keywords: hydrogen, solid fuels, pyrotechnic base, metal oxides and hydroxides, gas production **DOI:** 10.3103/S002713142006005X

INTRODUCTION

In the 1970s, a new direction appeared in technology, called "hydrogen energy" [1]. In hydrogen energy, hydrogen is considered as an energy carrier that can replace the existing natural energy carriers oil, natural gas, and coal. The main precondition is that there are practically unlimited raw materials reserves of hydrogen in nature (mainly water). In addition, during the combustion of hydrogen, water vapor is again formed and thus the water cycle in nature is maintained, which creates conditions for maintaining the natural environment in a balanced state. This sums up the unique properties of hydrogen.

Hydrogen as an effective fuel was widely used in rocket and space technology under the Shuttle»] program in the United States, in the Ariane launch vehicles in France, and in booster rocket blocks in India and Japan. The use of liquid hydrogen in the Saturn rocket and space system under the Apollo program allowed the United States to land a man on the Moon in the 1960s. In addition, in rocket and space technology, hydrogen was used in electrochemical generators (EGs) for the direct conversion of chemical fuel energy into electrical energy.

In the In Soviet Union, liquid hydrogen was used as rocket fuel and fuel for EGs under the Energia-Buran program, and also as fuel for the TU-155 experimental aircraft. Subsequently, projects were developed for rocket boosters with hydrogen-oxygen engines and hypersonic aircraft, where liquid hydrogen is simply needed not only as a high-calorific fuel but also as an effective coolant for bearing surfaces (the specific heat capacity of hydrogen is 6.5 times higher than that of kerosene).

The main methods for producing hydrogen are as follows: steam reforming of methane and natural gas, coal gasification, water electrolysis, pyrolysis, bio-technology, partial oxidation, etc. In the case of stationary systems for the generation of hydrogen, it is most energy-efficient to use electrolyzers, and in the case of autonomous steam accumulators, it is preferable to use hydro-reacting metals, which can have an electrochemical reaction with water, producing not only hydrogen but also electric energy and heat. In those cases when it is required to obtain a small amount of hydrogen (up to 10 m³) within a few tens of seconds, it is possible to use gas generators (GGs) based on solid fuel and pyrotechnic compositions in autonomous systems.

The main components of pyrotechnic compositions are (i) a substance that generates hydrogen and at the same time performs the function of a fuel, and (ii) an oxidizer that temporarily performs the function of fuel and an oxidizer that releases active oxygen during decomposition. Hydrogen with a temperature of 20°C and a purity of 99.6 to 100% can theoretically be obtained using hydrides and borohydrides of metals of a series of alkali metals and aluminum (AlH₄, LiBH₄, NaBH₄, KBH₄) as fuel. The reasons for their potential use are the high volume and mass density of hydrogen, as well as the soft conditions for its production. Thus, the density of hydrogen in potassium borohydride is 0.083 g/cm³; in sodium borohydride, 0.112 g/cm³; and in amine-borane, 0.145 g/cm³; this exceeds the density of liquid hydrogen (0.07 g/cm^3) [2]. As oxidants, it is preferable to use oxides such as CuO, Fe₂O₃, MnO₂, V₂O₅, and MoO₃, as well as metal hydroxides (Al(OH)₃, Ni(OH)₃ and H₃BO₃). Regular oxidizers made of nitrates and perchlorates of alkali metals in order to increase the burning rate of the compositions are used only in combined oxidizers.

Since the 1970s, considerable research has been carried out on the application of metal hydrides and borohydrides in hydrogen production.

In 1976, a solid fuel formulation based on AlH_3 was patented [3], where hydrogen was obtained by the reaction in the combustion mode (reaction temperature 3000 K, specific gas release of hydrogen 300 cm³/g):

$$AlH_3 + Fe_2O_3 \rightarrow 2Fe + Al_2O_3 + 3H_2.$$

Fuel of this composition has a low combustion rate, is not capable of rapid (explosive) transformation, and has a sufficiently high ignition temperature (at least 500° C).

Together with Fe_2O_3 , Al_2O_3 , In_2O_3 , and Co_2O_3 were used as oxidizing agents for borohydrides of the general formula Me(BH₄), the hydrogen temperature in this case ranges from 873 to 973 K. Cold hydrogen with the temperature from 453 to 460 K was obtained by the decomposition of AlH₃ [4, 5].

Research has been conducted on the use of hydrazine derivatives, in which hydrogen was produced by the combustion of hydrazine bisborane $N_2H_2 \cdot 2BH_3$ with the decomposition of diammonium diborane $H_2B(NH_3)_2BH_4$. In this case, the hydrogen yield ranged from 13.4 to 16.7 wt % with the hydrogen content ranging from 99.0 to 99.6 wt % [6].

Further studies were conducted using solutions of organic acids (formic, acetic, malic, citric, oxalic, ascorbic, tartaric, succinic, and tauric acids) [7, 8] or solutions of inorganic acids (hydrochloric, sulfuric, nitric, and phosphoric) [9–13]. The main disadvantage of these methods of obtaining hydrogen due to the interaction of metal hydrides and borohydride with acids is the difficulty of controlling the gas generation rate, since the process proceeds very rapidly (within 1–4 seconds).

At the same time, studies were carried out to obtain hydrogen from metal borohydrides (sodium borohydride, potassium borohydride) using various catalysts: ferric chloride [14, 15], chitosan [16], a catalyst based on cobalt boride [17, 18], sodium bicarbonate [19, 20], phosphoric acid [21, 22], a complex catalyst Co(II) Schiff Base [23], etc. The disadvantages of these methods of production are the formation of the reaction's by products that pollute hydrogen, harmful effects on the environment, and the high cost of catalysts.

In the 1990s, when the further study of solid fuel compositions required applied research, there was a need to develop test benches that would allow registering the nature of the changes in the pressure, as well as the temperature profile in two phases of the GG and the receiving capacity. A schematic diagram of such a test bench, developed and created by the specialists at Samara State Technical University, is shown in the figure.

The stand consists of a gas generator (1) and a gas duct (2) through which the generated gas enters the receiving tank (3). The material part is located in the armored cabin. The casing of the GG contains six connections in a thermocouple. Depending on the gas environment, chromel-copel, chromel-alumel, platinum-rhodium, and tungsten-rhenium thermocouples with a junction diameter of 20 μ m are used. They allow us to determine the nature of temperature changes along the GG axis and the speed of movement of the high-temperature zone of the main combustion reaction.

The pressure in the bottom part of the GG and in the prenozzle volume is recorded using pressure sensors (10). For the pressure sensors, the secondary devices consist of a tension unit (4) and an oscilloscope or computer (8); and for thermocouples, amplifiers (5)–(7). The oscilloscope is triggered when current is applied to the resistance bridge of the igniter. The readings of the thermocouple (9) and pressure sensor (10) are used to calculate the specific gas production.

An analysis of the patent and scientific literature showed that hydrogen production in most cases proceeds by the hydrolysis reaction of metal hydrides, which requires the organization of the subsequent storage and transportation of hydrogen. The authors of this article propose to use the solid fuel combustion method to produce hydrogen. In this case, it is necessary to additionally introduce a fuel binding agent in the solid fuel, which should serve as a source of hydrogen and create a porous coke residue that filters the combustion products throughout the entire volume of the GG.

Solid fuel formulations can be tested in two combustion modes: conductive and convective. Tests of cylindrical charges made of a solid fuel in the mode of end conductive combustion, when heat from the burning layer is transferred to the next one due to thermal conductivity, showed that the fuel burns at a low speed ranging from 0.5 to 3.0 mm/s. In order to increase the combustion rate of the compositions (which will also increase the consumption characteristics of the gas generator), it is proposed to use convective combustion. In this case, the fuel covers the entire section of the GG's chamber, the charge is ignited from the bottom end, the decomposition products of the composition pass either through the charge's pores or through the channels formed during the pressing of the through-passage, giving off heat to their surface and cooling to ambient temperature. This method of producing gases is called the mode of forced convection of combustion products (FCCP). This mode and the design of the GG were patented [24]. The use of such



Fig. 1. Schematic diagram of the test bench for determining the characteristics of solid fuel compositions: *1*, gas generator, *2*, gas pipe, *3*, receiving tank, *4*, amplifier of pressure sensors type LH, (5-7), amplifiers of thermocouple signal, *8*, oscilloscope H-117 or computer, *9*, thermocouple of the receiving tank, *10*, pressure sensor LH of receiving tank

a method makes it possible to increase the gas productivity of solid fuel compositions and provide conditions for the safe processing of gas production in the FCCP mode through the source substance.

EXPERIMENTAL

The aim of this paper is to study the effect of changes in the composition of the pyrotechnic base of solid fuels on the volume of hydrogen produced with a low gas temperature in the FCCP mode. KBH_4 was used as the fuel, since it is less hygroscopic than other

borohydrides and also less reactive with water. KBH₄ has the following properties:

$$T_{\rm m.} = 640^{\circ}{\rm C};$$

$$T_{\rm dec.} (\text{in nitrogen}) = 584^{\circ}{\rm C};$$

$$T_{\rm dec.} (\text{in hydrogen}) = 676^{\circ}{\rm C};$$

$$T_{\rm dec.} (\text{intensive}) = 680-700^{\circ}{\rm C};$$

$$\rho = 1.175 \text{ g/cm}^{3};$$

 $Q_{\rm form.} = 54.7 \text{ kcal/mol or } 1019.56 \text{ kcal/kg};$

MOSCOW UNIVERSITY CHEMISTRY BULLETIN Vol. 75 No. 6 2020

Composition, % S KBH ₄ /Fe ₂ O ₃	Specific gas	production, L/kg	Completeness of hydrogen release, $\%$ $W_{\rm pr.}/W_{\rm theor.}$	Burning rate,	Density	Porosity,	
	W _{pr.}	W _{theor.}		mm/s	$\rho_{theor.}$	$\rho_{pr.}$	%
35/65	286	290.5	100.0	8.24	2.32	1.24	45
40/60	280	332.0	84.3	6.67	2.18	1.18	46
45/55	250	373.5	67.0	5.85	2.04	1.10	46
50/50	140	415.0	33.7	5.62	1.90	1.05	45

Table 1. Test results of the pyrotechnic base of composition $KBH_4 + Fe_2O_3$

Table 2. Test results of the pyrotechnic base of the fuel composition $CuO + KBH_4$

$\frac{\text{Composition, \%}}{\text{KBH}_4/\text{CuO}_3} \frac{\text{Specific gas produced}}{W_{\text{pr.}}}$	Specific gas production, L/kg		Completeness of hydrogen release, $\%$ $W_{\rm pr.}/W_{\rm theor.}$	Burning rate, mm/s	Density	Porosity,	
	$W_{\rm theor.}$	$\rho_{theor.}$			$\rho_{\text{pr.}}$	%	
40/60	193.8	332.0	58.4	25.00	2.30	1.10	52
45/55	173.4	373.5	46.5	17.90	2.13	1.09	49
50/50	127.5	415.0	30.7	3.85	1.99	1.08	46
55/45	_	456.5	Attenuates	—	—	—	—

 $Q_{\text{dec.}} = 4264 \text{ kJ/kg}$. The thermal decomposition of KBH₄ proceeds according to the following equation [2]:

$$\text{KBH}_4 \rightarrow \text{K} + \text{B} + 2\text{H}_2 - 4264 \text{ kJ/kg}.$$

Oxides and hydroxides of metals (CuO, Fe_2O_3 , MnO_2 , MoO_3 , V_2O_5 , WO_3 , $Ni(OH)_3$, $Al(OH)_3$, H_3BO_3) were used as oxidizing agents. The thermal decomposition of the pyrotechnic base of solid fuels was studied using a Thermoscan-2 installation. When studying the pyrotechnic basis of solid fuels, the following parameters were estimated: density, specific gas production, combustion rate, and porosity.

At the first stage of the experimental research, the possibility of using the metal oxides (CuO, Fe_2O_3 , MnO_2 , MoO_3 , V_2O_5 , WO_3) as oxidizing agents was evaluated.

1. Pyrotechnic base of solid fuels of composition KBH_4/Fe_2O_3 .

Hydrogen production proceeds according to the KBH_4 oxidation reaction

$$KBH_4 + Fe_2O_3 \rightarrow KBO_2 + Fe + FeO + 2H_2 \uparrow.$$

The test results of this pyrotechnic base are shown in Table 1.

2. Pyrotechnic base of solid fuels of KBH/CuO composition.

Hydrogen production proceeds according to the KBH_4 oxidation reaction

$$\text{KBH}_4 + 2\text{CuO} \rightarrow 2\text{Cu} + \text{KBO}_2 + 2\text{H}_2 \uparrow$$
.

The test results of this pyrotechnic base are shown in Table 2.

3. Pyrotechnic base of solid fuels of $\text{KBH}_4/\text{MnO}_2$ composition.

Hydrogen production proceeds according to the reduction reaction

 $\text{KBH}_4 + \text{MnO}_2 \rightarrow \text{KBO}_2 + \text{Mn} + 2\text{H}_2 \uparrow$.

The test results of this pyrotechnic base are shown in Table 3.

4. Pyrotechnic base of solid fuels of the composition $\text{KBH}_4/\text{metal}$ oxide (MoO₃, V₂O₅, WO₃).

The test results are presented in Table 4.

According to the results of the tests carried out on the pyrotechnic base of solid fuels based on KBH₄ and metal oxides (CuO, Fe₂O₃, MnO₂, MoO₃, V₂O₅, WO₃), it can be seen that the pyrotechnic base with the use of MnO₂ as an oxidizing agent has the highest gas productivity; therefore, further studies on the influence of compacting pressure on the operating characteristics of the charge (specific gas production, combustion rate, density, porosity) were carried out on a pyrotechnic base consisting of KBH₄/MnO₂ in the ratio of 50/50. The test results are presented in Table 5.

From the analysis of the data given in Table 5, it can be seen that with an increase in the compacting pressure, there is a tendency the specific gas production to increase from 330 to 367 L/kg, the average combustion rate is 47.3 mm/s, the density increases to 0.98 g/cm³, and the porosity decreases by 13%; i.e., the filling factor of the chamber with fuel increases. Thus, among metal oxides, MnO₂ is the most promising oxidizing agent for KBH₄.

At the second stage, the possibility of using metal hydroxides as oxidizing agents was evaluated.

Composition, % KBH ₄ /MnO ₂	Specific gas production, L/kg		Completeness	Burning rate,	Density	Porosity,	
	W _{pr.}	W _{theor.}	$W_{\rm pr.}/W_{\rm theor.}$	mm/s	$\rho_{theor.}$	$\rho_{heor.}$	%
30/70	250	249.0	100.00	75.00	2.53	0.78	69
35/65	300	290.5	100.00	70.30	2.34	0.78	67
40/60	340	332.0	100.00	63.38	2.17	0.78	64
45/55	372	373.5	99.60	58.94	2.03	0.77	62
50/50	340	415.0	81.93	46.87	1.90	0.78	59
55/45	250	456.5	54.76	47.90	1.79	0.08	59
60/40	199	498.0	39.96	46.39	1.69	0.93	56
65/35	102	539.5	18.90	45.45	1.60	0.83	54

Table 3. Test results of the pyrotechnic base of the composition $KBH_4 + MnO_2$

Table 4. Test results of the pyrotechnic base of the composition $KBH_4 + MoO_3$, V_2O_5 , WO_3

Composition, %	Specific gas	production, L/kg	Completeness of hydrogen release, %	Burning rate, mm/s	
KBH_4/Me_xO_y	W _{pr.}	W _{theor.}	$W_{\rm pr.}/W_{\rm theor.}$		
55/MoO ₃	266.0	456.5	58.3	1.8	
55/V ₂ O ₅	—	—	Do not burn	—	
55/WO ₃	—	—	Do not burn	—	

Table 5. Test results of the pyrotechnic base of composition $KBH_4 + MnO_2$

Specific compacting pressure, MPa.	2.5	5.0	7.5	10.0	12.5	15.0
Specific gas production, L/kg	330.0 ± 10	344.2 ± 6	342.2 ± 8	354.4 ± 5	356.0 ± 10	367.0 ± 6
Burning rate, mm/s	46.61	45.84	42.00	45.35	53.90	48.52
Density, g/cm ³	0.73	0.80	0.83	0.85	0.85	0.98
Porosity, %	61	58	56	55	52	48
Completeness of hydrogen release, $\% W_{\rm pr.}/W_{\rm theor.}$	79.50	82.94	82.46	85.4	84.02	88.48

1. Pyrotechnic base of solid fuels of composition $KBH_4/Ni(OH)_3$.

Hydrogen production proceeds according to the reduction reaction

$$5\text{KBH}_4 + 6\text{Ni}(\text{OH})_3$$

$$\rightarrow 5\text{KBO}_2 + 2\text{Ni}_3\text{O}_4 + 9\text{H}_2 \uparrow.$$

The results are presented in Table 6.

As the amount of KBH_4 in the fuel increases the gas production decreases and the speed increases. In addition, at a component ratio of 45/55, the maximum release of hydrogen is observed, which indicates the participation of Ni(OH)₃ in the redox reaction.

In order to increase the gas productivity of compositions based on KBH_4/MnO_2 , the H_3BO_3 or $Al(OH)_3$ was introduced in them.

2. Pyrotechnic base of solid fuels of composition $KBH_4/MnO_2/H_3BO_3$.

The test results are presented in Table 7.

3. Pyrotechnic base of solid fuels of composition $KBH_4/MnO_2/Al(OH)_3$.

Hydrogen production proceeds according to the reduction reaction

$$3$$
KBH₄ + 4 Al(OH)₃

$$\rightarrow 2\text{Al}_2\text{O}_3 + 3\text{KBO}_2 + 12\text{H}_2$$

The test results are presented in Table 8.

The introduction of H_3BO_3 in the composition of the pyrotechnic base barely affects the gas productivity of the composition; however, the introduction of 10% H_3BO_3 has a significant effect on the combustion rate, causing its linear decrease by a factor of more than two due to the oxidizing agent that does not enter into the combustion reaction.

The introduction of 10 to 18% of aluminum hydroxide is accompanied by the gas production increasing by 20%, while the burning rate remains

Composition, % KBH ₄ /Ni(OH) ₃	Specific gas pr	oduction, L/kg	Completeness	Burning rate,	Density, g/cm^3	Porosity, %
	W _{pr.}	W _{theor} .	of hydrogen release, $\%$ $W_{\rm pr.}/W_{\rm theor.}$	mm/s	$(\rho_{\rm pr.})$	
45/55	460.0	541.6	84.98	37.1	1.90	46
50/50	450.0	567.7	79.30	59.1	1.83	53
55/45	397.8	594.0	67.00	57.1	1.74	50

Table 6. Test results of the pyrotechnic base of the fuel composition $KBH_4 + Ni(OH)_3$

Table 7. Test results of the pyrotechnic base of the fuel composition $KBH_4 + MnO_2 + H_3BO_3$ in the FCCP mode

Composition, % KBH ₄ /MnO ₂ /H ₃ BO ₃	Specific gas pr	oduction, L/kg	Completeness of hydrogen release, $\%$ $W_{\rm pr}/W_{\rm theor.}$	Burning	Density	Porosity,	
	W _{pr.}	$W_{\rm theor.}$		rate, mm/s	$\rho_{theor.}$	$\rho_{pr.}$	%
45/52.2/2.5	331.5	387	85.67	67.3	1.98	0.92	53
45/50/5	346.8	401	86.50	53.0	1.93	0.94	51
45/47.5/7.5	331.5	415	79.90	42.6	1.89	0.93	51
45/45/10	336.6	429	78.50	28.0	1.84	0.96	49

Table 8. Test results of the pyrotechnic base of the fuel composition $KBH_4 + MnO_2 + Al(OH)_3$ in the FCCP mode

Composition, %	Specific gas pr	oduction, L/kg	Completeness	Burning rate,	Density	Porosity,	
KBH ₄ /MnO ₂ /Al(OH) ₃	W _{pr.}	W _{theor.}	$W_{\rm pr.}/W_{\rm theor.}$	mm/s	$\rho_{theor.}$	$\rho_{\text{pr.}}$	%
45/52.5/2.5	341.7	384.2	89.0	52.10	2.0	0.87	56.5
45/50/5	357.0	395.4	90.3	54.60	1.99	0.85	56.0
45/47.5/7.5	359.9	406.6	87.8	64.00	1.97	0.86	56.0
45/45/10	377.4	417.8	90.3	63.30	1.95	0.87	55.0
45/40/15	418.3	438.1	95.5	20.35	1.90	0.87	54.0
45/35/20	234.0	462.4	50.6	5.10	1.87	0.88	53.0
45/25/30	180.0	507.4	3.5	No combustion	1.8	0.87	52.0

practically at the same level, a further increase in the $Al(OH)_3$ content leads to a decrease in gas production, and at 20% of $Al(OH)_3$ the combustion rate decreases by 12 times, and the gas production is 50% compared to the theoretical calculations.

The maximum gas productivity based on KBH_4 can be obtained by using Ni(OH)₃ as the oxidizing agent. In this case, the gas productivity is 460 L/kg.

CONCLUSIONS

In this article on the study of the possibility of developing a fuel formulation for the generation of hydrogen, it was noted that metal hydrides and oxides or hydroxides were mainly used to produce highpurity hydrogen both in the United States and Russia.

Hydrogen was produced from the pyrotechnic base of solid fuels in the FCCP mode. The tests have shown that in this mode, there is a multistage decomposition process of KBH_4 due to the pyrolysis of its residues upon contact with high-temperature slags after the passage of a combustion wave, which is confirmed by the completeness of its decomposition.

The most promising (from the point of view of the maximum value of the gas production and the completeness of decomposition of KBH_4) is the composition in which MnO_2 is used as the oxidizing agent. This composition has a combustion satisfactory rate for the FCCP mode, which is ten times higher than in the conductive mode.

The use of aluminum hydroxide and boric acid as oxidizing agents does not allow us to obtain a stable combustion in the FCCP mode. However, the addition of up to 15% of Al(OH)₃ to the composition based on KBH₄/MnO₂ leads to an increase in gas production by about 20%, while maintaining a constant burning rate and completeness of decomposition of the components with the release of hydrogen. It can be

assumed that at the first stage, MnO_2 as a peroxide compound reacts with KBH_4 , and at the second stage, the Al(OH)₃ is decomposed. The introduction of up to 10% H₃BO₃ does not affect the gas production but it more than halves the combustion rate.

Compositions containing $Ni(OH)_3$ have an undoubted advantage over compositions based on metal oxides, which allow us to increase the yield of hydrogen. $Ni(OH)_3$ is characterized by the maximum gas production. A preliminary assessment showed that a decrease in the Ni(OH)_3 content leads to an increase in the combustion rate, which requires more detailed studies.

In addition to the indicated direction of further research, it is necessary to study in more detail the composition based on MnO_2 and $Ni(OH)_3$ with a detailed study of the influence of the scale factor, the porosity of the charge, and the type of binder.

FUNDING

The study was carried out as part of the government's funding of the Faculty of Engineering and Technology, Samara State Technical University.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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