THE MANGANESE OXIDES DECOMPOSITION CATALYSTS FOR HIGHLY CONCENTRATED HYDROGEN PEROXIDE

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Abstract

The paper presents relatively simple but reliable laboratory method for the preparation of manganese oxides alumina supported catalysts for highly concentrated hydrogen peroxide decomposition. The number of batches of the pellet catalysts were prepared by simple wetness, impregnation method. In all cases the researched samples of the catalysts were impregnated with a precursor solution of potassium permanganate resulting in the formation of manganese oxide catalyst after the baking process. The primary assessment criterion performed after the preparation was percentage catalyst loading with respect to weight. The effect of various support kinds and the modifications of preparation process on the final properties of the catalyst has been evaluated. The activity and the durability have been assessed through the hot tests. The practical validation and application of the catalyst is its utilisation in the fixed catalyst bed in the rocket propulsion devices.

Keywords: heterogeneous catalyst, alumina, impregnation, hydrogen peroxide decomposition, HTP, hydrogen peroxide, hybrid rocket motor.

INTRODUCTION

This paper presents a simple and common method of preparation of alumina supported catalysts that are suitable for decomposition of highly concentrated hydrogen peroxide (98%+). It contains detailed description of all procedures connected to the methods of preparing in situ (in the laboratory at IoA) suitable quantities (batches) of heterogeneous supported catalysts for efficient decomposition of 98%+ HTP (High Test Peroxide). The prepared batches of the catalysts have been then explored and analyzed through their key features including design of appropriate composite catalyst bed, with functionality, durability, accuracy, and thereby providing some indication of their effectiveness at decomposing hydrogen peroxide of 98%+ class. The results of latter actions will be used to refine and improve the manufacturing process in the near future.

Highly concentrated (especially that of 98%+) hydrogen peroxide of HTP class, seems to be very promising substitute of hydrazine [1]. The medium is characterised by relatively high performance, non-toxic vapour and benign nature (practically non-irritant with low corrosivity). What more, the substance generates environmentally friendly decomposition

products, which make handling this rocket oxidizer considerably less difficult than NTO (dinitrogen tetraoxide) or RFNA (red fuming nitric acid). Thus, highly concentrated (typically above 80%) solutions of hydrogen peroxide, as a green propellant, are becoming currently very attractive as possible substitutes for hydrazine and dinitrogen tetraoxide. Over one generation back HTP used to be quite versatile rocket propellant but gradually was replaced by more toxic, and yet more effective, ones [2]. Nowadays, with growing concerns about the environment and personnel safety, HTP is becoming experience renewed interest. Especially 98%+ hydrogen peroxide of HTP class seems to have a great potential for replacement of toxic and corrosive propellants currently used for satellite applications. This is mostly due to the fact that the use of 98%+ HTP may in practice result in significant cost saving associated with the enormous simplification of the health and safety precautions necessary during the use (that is production, storage, handling, etc.) of the propellants. These advantages have a special relevance to low or medium thrust rocket engines or thrusters, where the above cost does not scale down proportionally to the engine size.

Several kinds of aluminium oxide (Al_2O_3), depending on its polymorphic phase and pellet size, were considered to be used as the catalyst supports. Some of them with a high specific surface area. It is assumed that relatively high specific area should enable a high dispersion of the active phase at high loadings. This might cause quite unique phase-support interactions. This should also result in the required catalytic efficiency − activity as well as durability. The latter feature is of great importance as the conditions during the process of 98%+ HTP decomposition are very harsh (oxidative environment and the temperature up to 1000°C). The main active phases include manganese oxides, which are typically used in hydrogen decomposition processes, doped with cobalt oxides, and in some cases possibly promoted with samarium oxide or lanthanum oxide.

The preliminary tests showed that the use of manganese dioxide (or oxides, Mn_xO_y) based catalysts on alumina support might be the benchmark technology for efficient, relatively cheap and controllable decomposition of 98%+ HTP. What more, the same type of catalyst support is currently utilised in satellite thrusters − alumina catalyst supports are used for manufacture of hydrazine decomposition catalysts [3].

DECOMPOSITION OF HIGH TEST PEROXIDE

Hydrogen peroxide in its purest form (100%) is inherently stable and storable liquid substance − and the solution of 98%+ HTP is too [4, 8]. However, if brought into contact with certain contaminants (e.g. transition metal salts or oxides) it rapidly decomposes. Therefore, stabilisers are normally added to the medium. Usually they act by complexing with any traces of transition metal ions present in the solution which would otherwise catalyse the decomposition of HTP. Typical stabilisers are sodium stannate (IV) (which hydrolyzes to tin (IV) hydroxide) and various phosphates [5].

The catalysts that have been typically utilised in practical applications for HTP decomposition usually have been prepared in the form of silver macroscopic screens (discs, gauze), activated with samarium compounds or/and diluted nitric acid. However, this type of catalysts have traditionally been used with HTP solutions of concentration not higher than 90%. HTP solutions concentrated above 90% (e.g. 98%+) cannot be used with silver catalysts. Such solutions cause strong catalyst sintering or even silver melting due to higher temperature of decomposition products [6].

It is well known fact now, that a catalytic solid is a material that facilitates a chemical reaction by lowering the activation barrier (Figure 1).
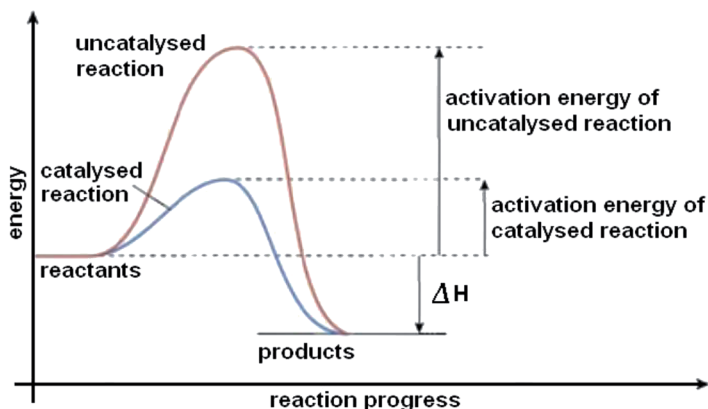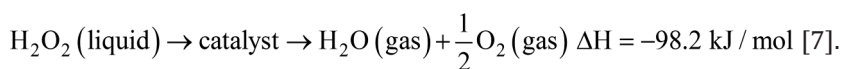
Figure 1. The activation energy variation as a function of reaction progress [Aut., 2015]

The catalysts for decomposition of concentrated hydrogen peroxide usually are comprised of a support and the catalytically active phase on it. It means that are of heterogeneous type of catalysts. In order to achieve highly efficient decomposition of 98%+ HTP, adequate for practical purposes – that is to be used in propulsion applications – several types of catalyst of various nature and geometric shapes have been investigated and experimentally evaluated in the decomposition chamber during the hot tests.

THE PREPARATION OF SUPPORTED CATALYSTS

It has been observed that manganese oxides as well as some other metal oxides of group VIII of the periodic table show reasonable activity in efficient decomposition of 98%+ HTP into desired hot gaseous products (steam and oxygen). The process is known as the catalytic decomposition of hydrogen peroxide. And with the use of suitable heterogeneous catalysts, about 2.8 MJ/kg of energy may be released during the process (for 100% solution):

$$H_2O_2\left(\text{liquid}\right) \rightarrow \text{catalyst} \rightarrow H_2O\left(\text{gas}\right) + \frac{1}{2}O_2\left(\text{gas}\right) \ \Delta H = -98.2 \text{ kJ / mol [7]}.$$

As the matter of fact, different catalyst supports, precursors and preparation conditions utilized may result in a wide range of properties of catalysts employed for the process of decomposition of HTP. Some of the most important properties of a particular catalyst for such purpose include; pore-size, pellet diameter, active phase content, doping and promotor behaviour and the number of calcinations performed – all of which will be investigated in the following research project through. However, to reach the goal, several different catalysts based on various kinds of alumina support have been synthesized and preliminarily characterized. The catalyst supports (bases) used in this study are comprised of low or high surface alumina (alpha, α or gamma, γ) or silica alumina derived compounds. The physical shapes of the supports particles (pellets) are either spheres or extrudates of a cylindrical geometry. The pellet diameters vary, but most of them are 1/8".

The eight different catalyst supports have been designated: base 01, base 02, base 03, …, and used for the manufacture of suitable batches of the test pellet catalysts.

The Table 1 provides the details of these catalyst supports (some basic properties as obtained from the manufacturers). And the Figure 2 presents the samples of the catalysts supports purchased for the investigation.

Table 1. The catalyst supports as obtained from the manufacturers

	Manufacturer	Product description
1	MaTecK GmbH	bimodal Al_2O_3-α, 3 mm pellets, surface area 250 $\frac{m^2}{g}$
2	MaTecK GmbH	Al_2O_3-α, 3 mm pellets, surface area 0.18 $\frac{m^2}{g}$
3	MaTecK GmbH	trimodal Al_2O_3-α, 6.35 mm rings, low surface area
4	Alfa Aesar GmbH	Al_2O_3-α, 4.8 mm spheres, low surface area
5	Alfa Aesar GmbH	Al_2O_3-SiO_2 (13%), 4.8 mm macro-porous spheres, low surface area
6	Alfa Aesar GmbH	bimodal Al_2O_3-γ, 3.2 mm pellets, high surface area
7	Alfa Aesar GmbH	activated Al_2O_3-metal basic, 3.2 mm pellets, surface area 90 $\frac{m^2}{g}$
8	Kurt J. Lesker Company	Al_2O_3-α, 1-2.5 mm Pieces, 99.99% pure

Figure 2. The samples of aluminium oxide supports used for the study [Aut., 2015]

THE PREPARATION METHOD

The alumina pellet supported catalysts batches have been manufactured using virtually the same, simple impregnation procedure with only the catalyst supports being changed. Additional samples have also been prepared, with utilization of extra elements as doping and/or promoting agents (additives). Some samples were prepared with slightly different impregnation procedure (e.g. impregnation under vacuum).

The first step of the catalyst preparation procedure was the selection of appropriate mass of the alumina support for its following calcination. The calcination process was performed in the electrical oven under controlled temperature (usually 600°C, 2 h). After weight determination, the support was subjected to the impregnating process with the appropriate solutions dissolved potassium permanganate (with extra additives in the case of some batches).

In some cases (a few batches) lanthanum nitrate hexahydrate was dissolved in distilled water and then impregnated onto the support prior to further impregnation with adequate metal salt. Next, the alumina was filtered and placed in the oven for drying and calcination.

Then, the mixed alumina oxide was ready for further impregnations of manganese or/and cobalt or other metals salts (if the batch was to be doped).

The general procedure may be described as follows:

1. Weigh pellets.
2. Calcination of pure pellets at 600°C (at 700°C for some batches) for 2 h.
3. Weigh pellets.
4. Agitation of the potassium permanganate solution (7 g $KMnO_4$ per 100 ml deionised water) with a magnetic stirrer at a raised temperature of 60°C, for 1 h.
5. Agitation of the pellets in the solution of $KMnO_4$ for 1 h, to remove air bubbles from the pellet's surface (some batches were subjected to the vacuum impregnation).
6. Calcination of the pellets at 600°C (at 700°C for some batches) for 2 h.
7. Weigh pellets when are cooled.

The samples of prepared catalysts provides the Table 2, whereas the Figure 3 present the general view of the samples.

Table 2. The total number of the catalyst batches prepared within the study for the further laboratory tests with 98%+ HTP

Batch	Name	Pre-calcination weight (g)	Post-calcination weight (g)	Final weight (g)	% wt loading		
1	20	80.486	75.175	79.211	5.37%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
1	32	89.116	83.085	90.844	9.34%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
2	41	79.082	78.958	82.611	4.63%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
3	2	80.137	73.738	82.225	11.51%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
4	3	79.986	79.929	82.882	3.69%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
4	40	70.052	69.958	74.896	7.06%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
5	15	80.079	80.069	81.418	1.68%	$KMnO_4$ + H_2O 7:100 Calcination 700°C	Mn_xO_y
5	8	–	80.052	82.965	3.64%	$KMnO_4$ + H_2O 7:100 3x (15min drying 100°C) calcination 700°C	Mn_xO_y
6	30	85.253	83.001	90.816	9.42%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
6	27	80.041	77.290	86.318	11.68%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
7	26	80.234	79.190	82.954	4.75%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
7	42	50.083	49.202	50.596	2.83%	$KMnO_4$ + H_2O 7:100 Calcination 600°C	Mn_xO_y
2	9	–	80.266	82.367	2.62%	$FeCl_3$ x $6H_2O$ + $KMnO_4$ + H_2O 1:6:100 3x (15min drying 100°C). calcination 700°C	Mn_xO_y. FeCl
6	11	80.192	77.171	80.428	4.22%	$FeCl_3$ x $6H_2O$ + $KMnO_4$ + H_2O 1:6:100 15 h 20°C, 0.1bara	Mn_xO_y, FeCl
4	18	70.362	70.345	71.106	1.08%	(0.1g) Sm_2O_3 + (6g) $KMnO_4$+ (100ml) H_2O	Mn_xO_y, Sm_2O_3
5	17	80.124	80.091	80.699	0.76%	(0.1g) Sm_2O_3 + (6g) $KMnO_4$+ (100ml) H_2O	Mn_xO_y, Sm_2O_3
6	21	81.062	77.216	81.689	5.79%	(0.15g) Sm_2O_3+ (6g) $KMnO_4$ + (100ml) H_2O	Mn_xO_y, Sm_2O_3
2	53	280.693	280.362	290.383	3.57%	(3g) $(NH_4)_2Cr_2O_7$ + (30g) $KMnO_4$ + (300ml) H_2O 20°C 24h vacuum drying 105°C 1h	$(NH_4)_2Cr_2O_7$, Mn_xO_y
4	39	80.073	79.898	83.197	4.13%	(2g) $(NH4)_2Cr_2O_7$+ (12g) $KMnO_4$ + (200ml) H_2O	$(NH_4)_2Cr_2O_7$, Mn_xO_y
7	10	80.051	79.009	82.542	4.47%	(1g) $(NH_4)_2Cr_2O_7$ + (6g) $KMnO_4$ + (100ml) H_2O	$(NH_4)_2Cr_2O_7$, Mn_xO_y
2	34	175.347	175.223	178.048	1.61%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
2	35	79.963	79.863	81.374	1.89%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
4	46	49.988	49.909	51.308	2.80%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
4	47	50.035	49.988	52.213	4.45%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
6	45	50.030	48.379	51.967	7.42%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
6	44	50.088	48.161	52.362	8.72%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$

Table 2. continued. The total number of the catalyst batches prepared within the study for the further laboratory tests with 98%+ HTP

Batch	Name	Pre-calcination weight (g)	Post-calcination weight (g)	Final weight (g)	% wt loading		
6	25	80.039	77.275	87.975	13.85%	(0.5g) $(CH_3COO)_2Co$ + (7g) $KMnO_4$ + (100ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
7	43	50.149	49.360	50.778	2.87%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
4	22	80.334	80.181	82.856	3.34%	(0.2g) Sm_2O_3 + (6g) $KMnO_4$ + (1g) $(CH_3COO)_2Co$ + (100ml) H_2O	Mn_xO_y $(CH_3COO)_2Co$, Sm_2O_3
5	33	–	171.819	178.229	3.73%	(0.13g) Sm_2O_3 + (6g) $KMnO_4$ + (0.9g) $(CH_3COO)_2Co$ + (100ml) H_2O	Mn_xO_y $(CH_3COO)_2Co$, Sm_2O_3
5	23	–	100.035	102.753	2.72%	(0.11g) Sm_2O_3 + (6g) $KMnO_4$ + (0.5g) $(CH_3COO)_2Co$ + (100ml) H_2O	Mn_xO_y $(CH_3COO)_2Co$, Sm_2O_3
5	31	86.615	86.431	87.883	1.68%	(0.2g) Sm_2O_3 + (5g) $KMnO_4$ + (1g) $(CH_3COO)_2Co$ + (100ml) H_2O	Mn_xO_y $(CH_3COO)_2Co$, Sm_2O_3
4	36	80.451	80.290	88.184	9.83%	(12g) $KMnO_4$ + (2g) $K_3[Fe(CN)_6]$ + (200ml) H_2O	Mn_xO_y, $K_3[Fe(CN)_6]$
4	37	80.322	79.975	90.025	12.57%	(12g) $KMnO_4$ + (2g) $K_3[Fe(CN)_6]$ + (200ml) H_2O	Mn_xO_y, $K_3[Fe(CN)_6]$
6	52	80.052	76.814	84.543	10.06%	(13g) $KMnO_4$ + (1g) $K_3[Fe(CN)_6]$ + (220ml) H_2O+ (1g) $La(NO_3)_2·6H_2O$ calcination, 2 h vacuum impregnation	$K_3[Fe(CN)_6]$, $La(NO_3)_2·6H_2O$, Mn_xO_y
1	51	80.132	74.732	79.615	6.53%	(13g) $KMnO_4$ + (1g) $La(NO_3)_2·6H_2O$ + (200ml) H_2O calcination, vacuum impregnation	$La(NO_3)_2·6H_2O$, Mn_xO_y
4	50	100.000	99.818	102.295	2.48%	(11g) $KMnO_4$ + (1.2g) $(CH_3COO)_2Co$+ (1g) $La(NO_3)_2·6H_2O$ + (200ml) H_2O	$(CH_3COO)_2Co$, $La(NO_3)_2·6H_2O$, Mn_xO_y
6	49	80.072	77.362	84.187	8.82%	(11g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (1g) $La(NO_3)_2·6H_2O$ + (200ml) H_2O	$(CH_3COO)_2Co$, $La(NO_3)_2·6H_2O$ Mn_xO_y
9	55	65.062	62.801	66.028	5.14%	(5g) $KMnO_4$ + (2g) $Co(NO_3)_2·6H_2O$ + (100ml) H_2O	$Co(NO_3)_2$, Mn_xO_y
6	54	104.852	101.549	107.210	5.57%	Pre - caltination = impregnation (1.05g) $La(NO_3)_3·6H_2O$, post - caltination = (14g) $KMnO_4$ + (200ml) H_2O vacuum impregnation	$La(NO_3)_3·6H_2O$, Mn_xO_y
2	57	–	–	174.869	2.00%	Mn_xO_y + CrO on alpha alumina 3mm pellets	–
1	58	–	–	60.351	8.00%	Mn_xO_y + LaCrO3 on gamma alumina	–
4	59	–	–	269.190	3.50%	Mn_xO_y + CoO on alpha alumina pellets	–
5	16	80.322	80.327	81.538	1.51%	(6g) $KMnO_4$ + (1g) $(CH_3COO)_2Co·4H_2O$ + (100ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
8	38	100.055	100.008	101.000	0.99%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$
8	29	100.000	99.935	105.181	5.25%	(0.3g) Sm_2O_3 + (5g) $KMnO_4$ + (1g) $(CH_3COO)_2Co$ + (100ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$, Sm_2O_3
10	48	50.015	49.995	50.700	1.41%	(12g) $KMnO_4$ + (2g) $(CH_3COO)_2Co$ + (200ml) H_2O	Mn_xO_y, $(CH_3COO)_2Co$

Figure 3. Ready for hot tests manganese oxides ceramic based catalysts in the form of pellets
(some of them doped and/or promoted) [Aut., 2015]

THE RESULTS AND APPLICATIONS

Tens of samples of pellet catalysts were prepared and 8 different supports were used for this research task. Most of the support samples were impregnated only with manganese oxides, using virtually the same procedure. In order to avoid too many hot tests, the selection of representative samples was made. Two samples for each support, one with minimum and the other one with maximum value of the active phase content, were selected. In case of two of the eight supports, just one sample was available.
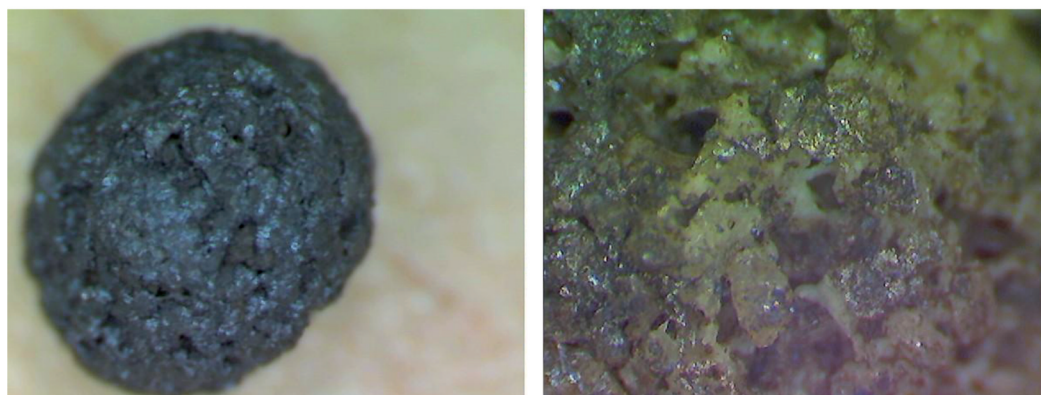

Figure 4. A single Mn_xO_y-Co_xO_y/Al_2O_3 catalyst pellet of 3 mm in diameter (left, 50x magnification) and its surface structure with visible cristals of active phase (right, 250x magnification) after the hot tests [Aut., 2015]

Some of the tested catalysts have shown very high efficiencies even in highly unfavourable conditions. The Figure 5. provides an example of successful utilization of this type of catalyst in a research mono-propellant rocket motor. The catalysts based on Al_2O_3-SiO_2 (13%), 4.8 mm macro-porous spheres, low surface area as the support were able to survive tens of seconds of such tests without visible symptoms of shattering or washing out (Figure 4). The temperature during the test was at the constant level between 950 to 1000°C.
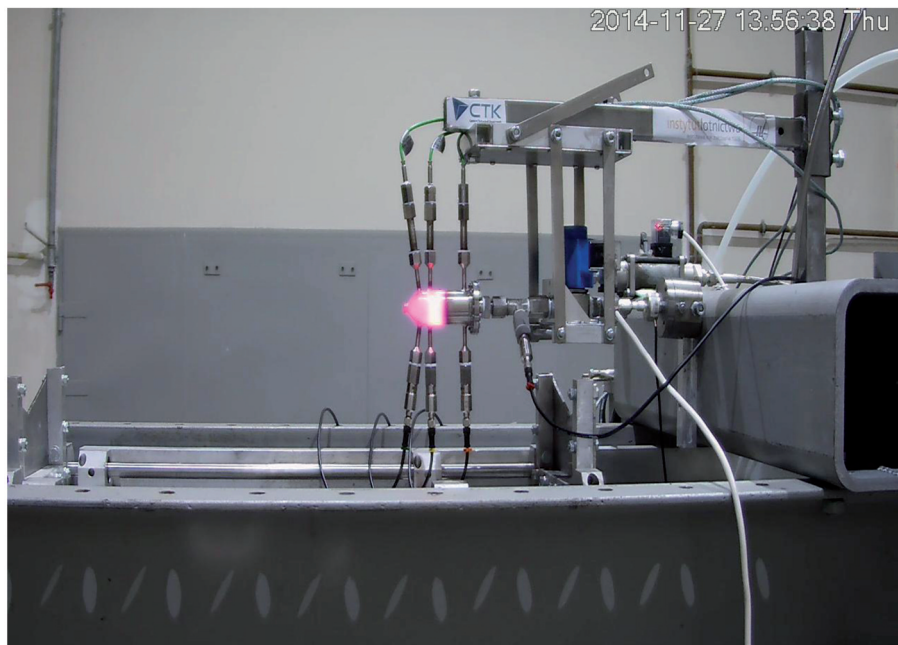
Figure 5. The hot test of the prepared catalysts with 98%+ HTP in a research mono-propellant rocket motor [9]

CONCLUSIONS

The prepared catalysts have appeared to be highly effective in this process of 98%+ decomposition. The final validation was done throughout appropriate hot tests. All the tested catalysts decomposed 98%+ HTP very efficiently at ambient initial temperature of the catalyst bed. The support no. 05 (Al_2O_3 with 13% SiO_2, 4.8 mm macro-porous spheres, low surface area from Alfa Aesar GmbH) was identified experimentally as the best type of catalytic support (in terms of robustness) from the selected ones for the catalyst preparation for 98%+ HTP decomposition.

The manganese oxides on alumina pellets catalysts should provide higher contact surfaces than e.g. metal grid or silver gauzes catalysts. This is due to the fact that their surface is highly developed – at least several tens of square meters per gram. This means that they may require only a fraction of residence time for decomposition reactions of HTP compared to e.g. platinum or silver grid catalysts. Thus, relatively small catalyst volumes may be needed, making the catalytic system with a manganese oxides based catalyst bed very cost effective. Also, the inlet concentration of HTP should not affect the amount of catalyst required or the design of the system. Additionally, the ceramic material of the support could perform as heat reservoir to absorb abundant heat released from the 98%+ HTP decomposition process.

ACKNOWLEDGMENTS

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WPŁYW KATALIZATORÓW TLENKU MANGANU NA ROZKŁAD KATALITYCZNY NADTLENKU WODORU

Streszczenie

Artykuł opisuje stosunkowo prosty i niezawodny sposób laboratoryjnego wytwarzania katalizatorów rozkładu wysoko stężonego nadtlenku wodoru. Każdą partię katalizatorów wytworzono w procesie impregnacji i spiekania. Tlenek glinu w postaci pelletów impregnowano w roztworze nadmanganianu potasu, następnie poddawano go spiekaniu i otrzymywano gotowy katalizator. Katalizatory po procesie poddawano ocenie zawartości fazy aktywnej, wpływu prekursorów oraz modyfikacji sposobu wytwarzania na jego trwałość.

Słowa kluczowe: katalizator heterogeniczny, tlenek glinu, impregnacja, nadtlenek wodoru, rozkład nadtlenku wodoru, hybrydowy silnik rakietowy.