



Developing the Physicochemical Foundations to Form Heterogeneous Rocket Propellants Using the Loading Method

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Received by the editorial staff on 28 October 2016

Reviewed and verified version received on 10 January 2017

DOI 10.5604/01.3001.0010.1571

Abstract. The physicochemical foundations are being developed to form propellants using the loading method. The advantages of this method include the possibility of achieving a greater packing density of the particulates, no requirement for the use of costly equipment, and the absence of mechanical operations that could pose a risk of fire or explosion. The determined values were dimensions, helium and bulk density, closed pore content, (the content of intergranular spacing and open pore), and the thermal stability of ammonium perchlorate (AP) granules originating from various sources. The obtained materials differed in terms of size, sphericity, bulk density, closed pore density, (the content of intergranular spacing and open pore). The granulate having the greatest sphericity and bulk density was determined based on tests.

The measurements, sphericity and density of aluminium dust originating from various sources were established. The pot life of the prepolymer cross-linking agent (diisocyanate) system was specified. Cross-linking agents with a pot life exceeding 3 hours at a temperature of 70°C were selected. Based on the tests, materials were selected for the formation of heterogeneous rocket propellants using the loading method. The propellant samples created using the loading method were characterised in terms of their physicochemical parameters.

Keywords: chemical technology, fuel formation, heterogeneous propellants, cross-linking, loading method

1. INTRODUCTION

A heterogenous rocket propellant, before the cross-linking process, is a mixture of oxidiser, flammable substance, binder, plasticiser and technological additives improving the performance parameters of the product. The mixture, following curing, has the properties of a solid body.

The production of solid rocket propellants is based on complex chemical and physical processes. The methods employed include pressing, extrusion, casting, and the loading method [1-4].

The loading method was selected to form heterogeneous rocket propellants. The advantages of this method include the possibility of direct formation of the propellant in the engine chamber, the possibility of obtaining a greater packing density, and the absence of mechanical operations that could pose a risk of fire or explosion. Some limitations of this method include the durability of the particulate granules under load.

1.1. Foundations for forming propellants using the loading method

A schematic of the laboratory system for the loading method is presented in Figure 1. Vessel I contained all the solid components in appropriately selected proportions. The grate in vessel I was used to hold the particulate granules. A liquid mixture of the specified composition, also containing all the components soluble in it, was then poured into vessel II. All the components of the system were grounded to protect them against the effects of static electricity. The resulting system was placed inside a pressure chamber with the ability to increase and decrease the pressure. The first stage reduced the pressure to 100 kPa for 2.5 hours, to degas the materials in vessels I and II. For the second stage, the pressure was increased to an overpressure of 1 MPa for 15-30 minutes. Any neutral, non-combustible gas, such as nitrogen, can be used as the gaseous medium. As the pressure increases, the liquid components were transported to the porous matrix formed by the solid materials in vessel I. The final step was to condition the material in vessel I at a temperature of 70°C for 1 week.

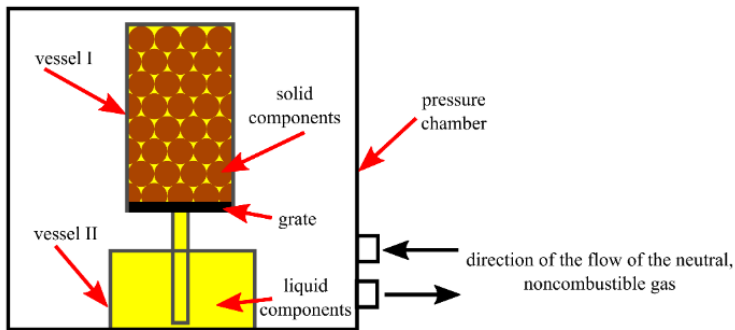


Fig. 1. Scheme used for propellant sample formation under the loading method

The content of the solid components in the propellant depends on the packing density of the particulates. By approximating the shape of the particulates to spheres of equal dimensions, the highest packing density (φ_{\max}) is obtained in a Face Centered Cubic (FCC) structure and Hexagonal Close Packed (HCP) structure, with $\varphi_{\max} = 0.7405$ [5]. The arrangement of spheres is presented schematically in Figure 2 A. The granules of the solid bodies create free spaces (gaps), which are indicated in yellow. Other granules with suitably small diameters can be introduced into the free spaces. Such granules create new free spaces, in which one can place even smaller granules. This is presented schematically in Figure 2 B.

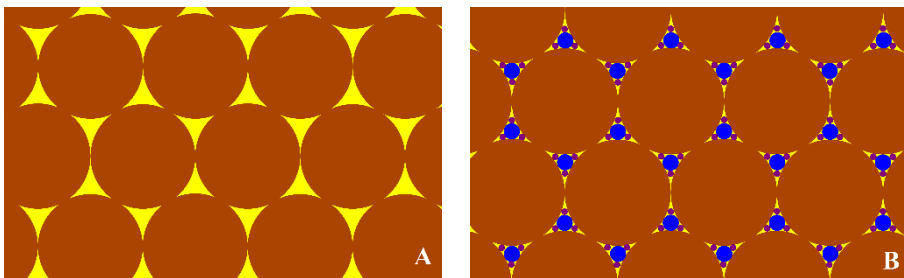


Fig. 2. The scheme for maximum packing of granules with equal diameters (A) and three different diameters (B)

The highest packing density can be obtained by selecting the appropriate ratios of diameters and quantities of granules in systems composed of granules with 2-3 different diameters. The maximum packing density in the FCC structure is $\varphi_{\max} = 0.8916$, with the ratio of the radii of the three granules being 1:0.4142:0.2929 (granule quantity ratio 4:3:24). In the HCP structure, the maximum packing density is lower ($\varphi_{\max} = 0.8556$) for the three granule ratio 1:0.6330:0.3050 (quantity ratio 6:2:15) [5].

These values assume an ideal arrangement of spheres, while in practice the packing densities are smaller, at $\varphi = 0.60-0.64$ for monodispersed spheres.

The obtained heterogeneous rocket propellants have the following composition: ammonium perchlorate (AP) 65-75%_{mas}, Al 10-20%_{mas}, binder 7-15%_{mas}, plasticiser 2.5-4.0%_{mas}, cross-linking agent 0.05-0.15%_{mas}, iron oxide 0.5-3.0%_{mas}, other additives 0.005-5%_{mas} [6]. Taking into account the ratio of the binder (hydroxyl-terminated polybutadiene (HTPB)), plasticiser (dioctyl adipate (DOA)), cross-linking agent (2,4-toluene diisocyanate (TDI) and iron compound(2,2'-bis(ethylferrocenyl)propane – catocene) [6] and their density, we can calculate the anticipated rocket propellant composition on the basis of the packing density. The mass composition is presented in Table 1.

Table 1. Composition of the rocket propellant calculated based on maximum packing for equal diameter granules

Component	Content/ % _{mas}	
	$\varphi = 0.60-0.64$	$\varphi_{\max} = 0.7405$
AP	75.24-78.27	85.25
HTPB	11.21-12.77	7.61
DOA	6.41-7.30	4.35
catocene	3.87-4.41	2.63
cross-linking agent	0.24-0.27	0.16

The objective of this work is to develop the foundations for forming heterogeneous rocket propellants using the loading method. The advantages of this method are the possibility of higher packing densities for the particulates in the form and the absence of mechanical operations. In the first stage of the tests, the components used in rocket propellants are characterised, and then the propellant samples formed using the loading method on a laboratory scale.

2. MATERIALS AND RESEARCH METHODS

2.1. Materials

Solid and liquid components, as used in heterogeneous rocket propellants, were used for the tests. The materials for the samples were collected from various sources, from companies and scientific institutions dealing with rocket propellants. Granular ammonium perchlorate (AP) was used as an oxidiser, and the samples were marked: CI, CM, UI, UM, and 7M. The code 'C' means that the granulate comes from recycled rocket propellants. The aluminium dust came from three different sources, and was marked: I, APS-7 and GS-32.

Hydroxyl-terminated polybutadiene (HTPB) was used as the prepolymer, and marked as HTLO and HTPB(U).

The vitrification temperatures of both polymers were approx. -70°C . Dioctyl adipate (DOA) supplied by Boryszew S.A. was used as the plasticiser.

DOA was characterised by a purity of 99.5% and density within the range $\rho = 0.922\text{--}0.930\text{ g}\cdot\text{cm}^{-3}$. The cross-linking agents were: isophorone diisocyanate (IPDI) – Sigma Aldrich with 98% purity ($\rho = 1.05\text{ g}\cdot\text{cm}^{-3}$), 2,4-toluene diisocyanate (TDI) – Sigma Aldrich with 95% purity ($\rho = 1.21\text{ g}\cdot\text{cm}^{-3}$), 4,4'-diphenylmethane diisocyanate (MDI) – Sigma Aldrich with 98% purity ($\rho = 1.18\text{ g}\cdot\text{cm}^{-3}$) and dimeryl diisocyanate (DDI).

The other components used in forming the propellant samples were: 2,2'-bis(ethylferrocenyl) propane (catocene) – H-isophthaloyl-bis(2-methylaziridine) (HX-752) – an agent increasing adhesion between solid and liquid bodies.

The propellant samples were formed using the loading method. The samples obtained this way had the following dimensions: diameter – 8.7 mm, height – 9.9 mm and volume 0.6 ml. Argon was used as the gaseous medium during sample formation.

2.2. Research methods

The analysis of the geometry and surface texture of the ammonium perchlorate was conducted using optical microscopes: Smart Pro 5MP by Delta Optical and PZO SK14. The aluminium geometry was evaluated using a PZO SK14 optical microscope. The rheological properties of the liquid components were determined using a Brookfield DV2T-HB viscometer, and a small sample adapter, the SC4-13R(P) sample chamber and the SC4-21 spindle were used.

The analysis of the thermochemical properties was conducted using the SDT Q600 device by Thermal Analysis Instruments. Measurements were taken in open aluminium vessels, for the temperature range $T = 25\text{--}550^{\circ}\text{C}$ and a temperature increase speed of $\beta = 5^{\circ}\text{C}\cdot\text{min}^{-1}$.

Helium density analysis was carried out using the AccuPyc 1330 helium densitometer at a temperature of 28°C . The samples had a volume of 0.6 cm^3 . The bulk densities of the materials were determined for a volume of 5 cm^3 . The material was loosely poured and then shaken.

The hardness of the propellant samples was measured using a durometer (scale A) by C.V. Instruments, in line with ASTM D 2240 [7]. The measurements were taken at a temperature of $23.0\pm 2.0^{\circ}\text{C}$.

3. RESULTS AND DISCUSSION

3.1. Characteristics of ammonium perchlorate

All the samples of ammonium perchlorate were characterised by an irregular external and internal structure.

Figure 3 presents images of the AP CM and CI surface texture. Granules submerged in silicone oil feature dark areas which indicate air enclosed in the closed pores.

These dark patches also indicate pores in the structure that are, however, out of focus. Similar images were observed for the remaining AP samples.

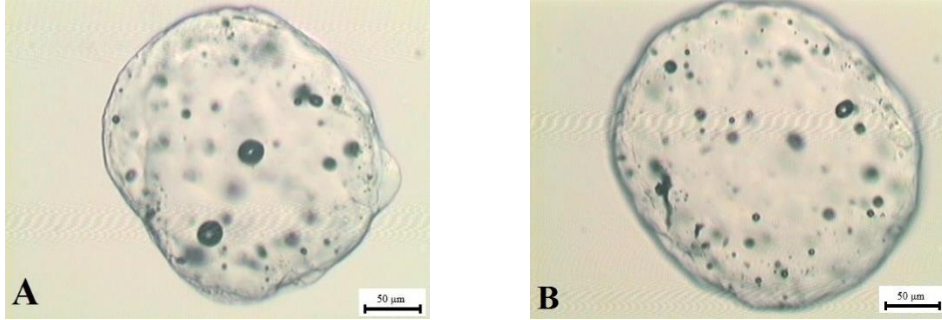


Fig. 3. The surface texture of AP CM (A) and CI (B) in silicone oil

Two perpendicular diameters, sphericity, helium and bulk density of all the AP samples were measured. The degree of sphericity was defined as a ratio of the measured diameters to their average value. It was assumed that the granules were spherical in shape, if the sphericity degree parameter (k) was lower than or equal to 10%. In addition, the percentage content of closed pores (P_z), intergranular spaces and open pores (P_o) was calculated in line with the formulas (1) and (2):

$$P_z = \frac{\rho_{AP} - \rho_{He}}{\rho_{AP}} \cdot 100\% \quad (1)$$

$$P_o = \frac{\rho_{AP} - \rho_{nAP}}{\rho_{AP}} \cdot 100\% - P_z \quad (2)$$

where: ρ_{AP} – AP density as indicated in the reference literature, ρ_{He} – density determined using the helium densitometer, ρ_{nAP} – bulk density of AP.

Table 2 summarises the above parameters. AP UI and UM are characterised by the largest diameters; however, the standard deviation indicates the substantial non-homogeneity of the granulates. AP 7M is characterised by the smallest diameter, and was obtained probably by the milling of granular AP. The highest sphericity was obtained for AP CI, at more than 93%. The densities determined by the helium densitometer correspond, within the error tolerance, to those stated in the reference literature ($\rho_{AP} = 1.95 \text{ g} \cdot \text{cm}^{-3}$ [8]). The best values of bulk density were observed for AP granules with the highest sphericity.

The percentage content of closed pores was very low (below 0.8%) for all the materials. Based on the drawn AP characteristics, AP CI was selected for the formation of propellant samples using the loading method, due to the high bulk density and the highest granule sphericity.

On the basis of the values obtained for AP CI, a theoretical composition for a heterogeneous propellant was determined: 81.3%_{mas} AP, 9.6%_{mas} HTPB, 5.5%_{mas} DOA, 0.2%_{mas} TDI, and 3.3%_{mas} catocene.

Table 2. Size, sphericity, density and the pore content for granules of ammonium perchlorate (* milled granules)

AP	$d/\mu\text{m}$	$k/\%$	$\rho_{\text{He}}/\text{g}\cdot\text{cm}^{-3}$	$\rho_{\text{nAP}}/\text{g}\cdot\text{cm}^{-3}$	$P_d/\%$	$P_o/\%$
CI	256±33	93.34	1.9361±0.0035	1.3209±0.0099	0.71	31.55
UI	318±55	34.45	1.9444±0.0024	1.2704±0.0065	0.29	34.57
CM	243±37	72.13	1.9428±0.0033	1.3219±0.0116	0.37	31.84
UM	310±57	58.62	1.9453±0.0029	1.1728±0.1214	0.24	39.61
7M	7±2	*	1.9434±0.0039	0.6849±0.0129	0.34	64.53

Thermogravimetric (TG) measurements were taken to determine the thermal stability of the AP granules. Figure 4A presents the mass derivatives (temperature of all samples), and Figure 4B presents the dependence of heat flow on temperature (DSC/TG curve).

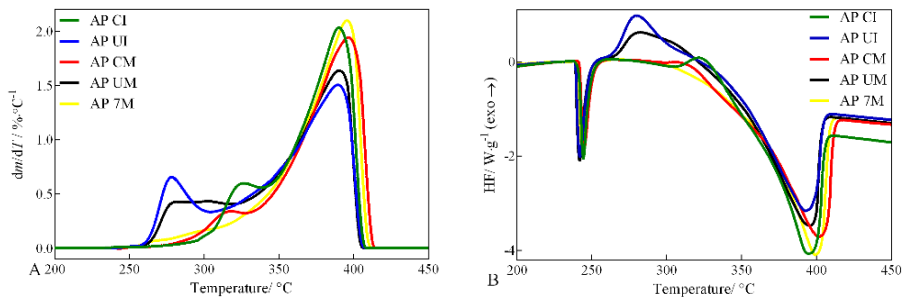


Fig. 4. A – mass derivatives (temperature), B – dependence of heat flow on temperature for different samples of AP (CI, UI, 7M, CM, UM)

The DSC/TG curve indicated three processes. The first was an endothermic process at $T = 243.4^\circ\text{C}$ with process enthalpy $\Delta H = 113.6 \text{ J}\cdot\text{g}^{-1}$, which was not associated with any decrease in mass, but corresponded to a transition from an orthorhombic form to a cubic one [9]. For all the tested samples, the decrease in mass was a two-stage process. The first stage, at a temperature of 250–330°C, corresponds to the initial, low-temperature decomposition of AP granules, which can be seen in the DSC/TG curve as an exothermic process.

It was observed that this process for AP from recycled propellants (AP CI and CM) took place at a temperature above 310°C, and was characterised by an approx. 15% decrease in mass and process enthalpy $\Delta H \approx 160 \text{ J}\cdot\text{g}^{-1}$. For AP UI and UM the process was observed below $T = 285^\circ\text{C}$, the decrease in mass was approx. 20%, and $\Delta H \approx 220 \text{ J}\cdot\text{g}^{-1}$.

The second stage, evident at a temperature above 330°C, corresponded to the high-temperature decomposition of AP granules – an endothermic process. This was mainly AP sublimation due to measurements being taken in open vessels [9, 10]. No interrelation between AP origin and the parameters of endothermic processes were observed. All samples of ammonium perchlorate maintained a similar level of thermal stability.

3.2. Characteristics of aluminium

Based on the preliminary analysis using an optical microscope, it was observed that the aluminium created agglomerates. To break down the agglomerates and determine the dimensions and sphericity, a mixture of Al and HTPB was made and placed in a ball mill, in which the components were mixed for 20 minutes at a frequency of 30 Hz. See Fig. 5 for the resulting images. The large darker areas are the result of air bubbles. Mixing in the ball mill broke down the Al agglomerates, for which dimensions and sphericity were determined for all types of Al.

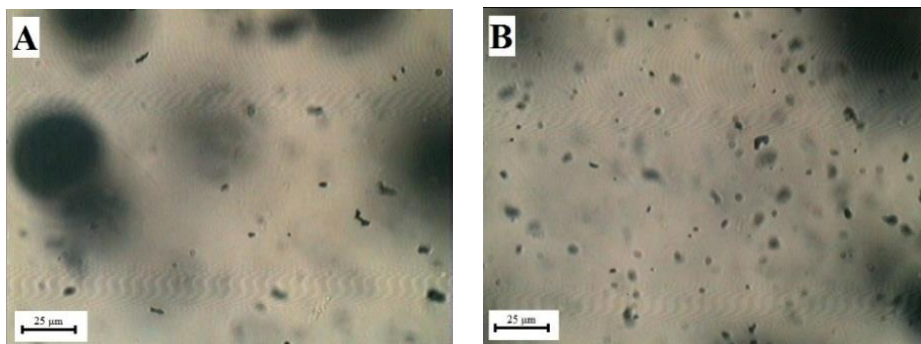


Fig. 5. The aluminium I (A) and APS-7 (B) images obtained by ball mill mixing with HTPB

Table 3 presents the results in terms of dimensions, sphericity and density, determined using the helium densitometer. The dimensions of the three types of aluminium were consistent within the deviation margin and were less than $10 \mu\text{m}$. The sphericity of particulates was low, at a maximum of 30%. The density of all samples was consistent with that presented in the reference literature, which is $\rho_{\text{Al}} = 2.67 \text{ g}\cdot\text{cm}^{-3}$ [11].

Ensuring the appropriate dispersion of aluminium in HTPB can facilitate the introduction of aluminium into the free spaces between the AP granules, together with a mixture of liquid substances (HTPB, DOA, etc.).

Table 3. Size, sphericity and density of aluminium (I, APS-7, GS-32)

Al	$d/ \mu\text{m}$	$k/ \%$	$\rho_{\text{He}}/ \text{g}\cdot\text{cm}^{-3}$
I	4.4 ± 1.7	30	2.679 ± 0.013
APS-7	3.6 ± 1.7	22	2.676 ± 0.009
GS-32	7.9 ± 3.1	29	2.688 ± 0.014

3.3. Rheological properties of polybutadiene + cross-linking agent

The rheological properties of two-component systems composed of polybutadiene and a cross-linking agent were determined during the cross-linking process. The amount of the cross-linking agent was determined using the formula [12]:

$$W_{\text{siec}} = \frac{NCO}{OH} \cdot EW_{\text{siec}} \cdot \left(\sum \frac{W_{\text{scOH}}}{EW_{\text{scOH}}} \right) \quad (3)$$

where: W_{siec} – amount of cross-linking agent, NCO/OH – ratio of isocyanate groups (NCO) to hydroxyl groups (OH), EW_{siec} – equivalent mass of the cross-linking agent, W_{scOH} – amount of liquid components with OH groups, EW_{scOH} – equivalent mass of liquid components with OH groups. For HTPB(U) the equivalent mass was $EW_{\text{scOH}} = 1220$. The equivalent masses of individual cross-linking agents were: $EW_{\text{siec}} = 87$ – TDI, $EW_{\text{siec}} = 111$ – IPDI, $EW_{\text{siec}} = 125$ – MDI, $EW_{\text{siec}} = 294$ – DDI.

The typical ratio of viscosity to time is shown in Figure 6. The experimental data were described with the general formula:

$$\eta = A \cdot e^{\frac{B}{t+t_u}} \quad (4)$$

where: η – viscosity, A , B – constant parameters, t – time, t_u – time for the complete cross-linking of the mixture.

The viscosity limit for which it was possible to cast the propellant was $\eta_{\text{max}} = 1.5$ kPas [13]. The time taken for the tested mixture to reach this level of viscosity is sometimes referred to as pot life (t_{pl}). The times for the complete cross-linking of the mixture and the pot lives of various two-component systems are presented in Table 4.

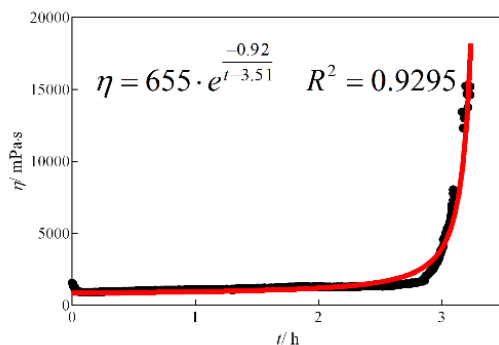


Fig. 6. Dependence of viscosity for curing time for HTPB(U)+TDI(M) system at a temperature of 70°C

Table 4. Time to complete curing of mixture (t_u) and pot life (t_{pl}) for binary systems HTPB(U)+curing agent determined during cure

Cross-linking agent		$\frac{NCO}{OH}$	$T/^\circ C$	t_{pl}/h	t_u/h
Type	Quantity/ %				
IPDI	3.05	0.35	25	> 4.5	> 4.5
	3.18	0.36	70	> 4.0	> 4.0
	7.58	0.90	70	1.85	5.97 ± 0.03
MDI	8.58	0.91	70	0.24	0.52 ± 0.06
DDI(M)	3.24	0.14	70	> 10.50	> 10.50
TDI	2.97	0.43	70	5.36	5.41 ± 0.01
	3.35	0.49	70	4.80	5.12 ± 0.02
	6.41	0.96	70	1.74	2.00 ± 0.04
TDI(M)	3.20	0.46	70	3.39	3.51 ± 0.01

Greater amounts of the cross-linking agent (and therefore greater ratios of NCO groups to OH groups) result in shorter pot lives. The system with MDI was characterised by the shortest pot life, at 0.24 hours. Therefore, MDI is not suitable for propellant forming using the loading method. Other cross-linking agents are characterised by long pot lives, exceeding 1.7 hours at a temperature of 70°C. It is planned to use TDI, DDI and IPDI in the forming of the solid rocket propellants using the loading method.

3.4. Characteristics of samples obtained using the loading method

Ten samples of propellants of various compositions were obtained using the loading method. The simplest mixture utilised three basic components – oxidiser, prepolymer and cross-linking agent.

In addition, one sample was obtained of propellant with a composition similar to the rocket propellants.

The composition of the individual propellant samples and their designations are presented in Table 5. Granules of AP CI, HTPB(U) and Al I were used in the samples. Another type of material was marked with an asterisk, i.e. crystalline ground AP was used in PK10T, and HTLO instead of HTPB(U) in PH03T. The digits indicate the content of the cross-linking agent (values multiplied by 10 and specified with an accuracy of a unit), while the last letter indicates the type of diisocyanate – I for IPDI and T for TDI.

Table 5. Composition of propellant sample formation using the loading method (* other type of material)

Symbol	Mass composition/ % _{mas}				
	AP	Al	HTPB	The cross-linking agent	Additives
P07I	75.4	-	23.9	0.7	-
P13I	82.9	-	15.8	1.3	-
P02T	78.1	-	21.7	0.2	-
P07T	78.3	-	21.0	0.7	-
P08T	75.7	-	23.5	0.8	-
P14T	75.1	-	23.5	1.4	-
PA05T	65.4	15.4	18.7	0.5	-
PK10T	70.5*	-	28.5	1.0	-
PH03T	59.94	13.68	13.62*	0.31	7.70% ADO, 0.03% HX-752, 4.72% catocene

Following the conditioning of the propellant samples for 7 days at 70°C, images of the propellant sample profiles were recorded using an optical microscope to evaluate the filling of the free spaces with liquid components.

Sample images for P07T are presented in Figure 7A, and for PH03T in Figure 7B.

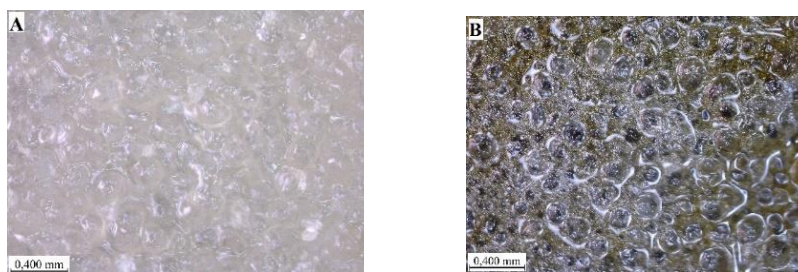


Fig. 7. Images of the profile propellant samples for P07T (A) and PH03T (B)

Liquid components filled all the free spaces of the porous matrix created by AP – such behaviour being observed for all the tested samples. In PH03T the Al was evenly distributed and no agglomerates were observed.

The propellant sample hardness (H) results are presented in Figure 8. The dashed red line marks the minimum and maximum values of propellant hardness, as indicated in the reference literature, in the range $H = 40\text{-}70^\circ\text{Sh}$ (scale A, temperature $23.0 \pm 2.0^\circ\text{C}$) [14, 15]. The introduction of 0.7% IPDI or 0.2-0.8% TDI is not enough to obtain propellant samples with hardness values similar to the reference literature data. The introduction of 1.3% IPDI and 1.4% TDI resulted in samples with a hardness of approx. 60°Sh . The PA05T and PH03T samples were characterised by a hardness below 40°Sh . The amount of introduced cross-linking agent largely influences the hardness. Therefore, the appropriate amount of the cross-linking agent should be introduced for propellant samples to have hardness values similar to those in the reference literature data.

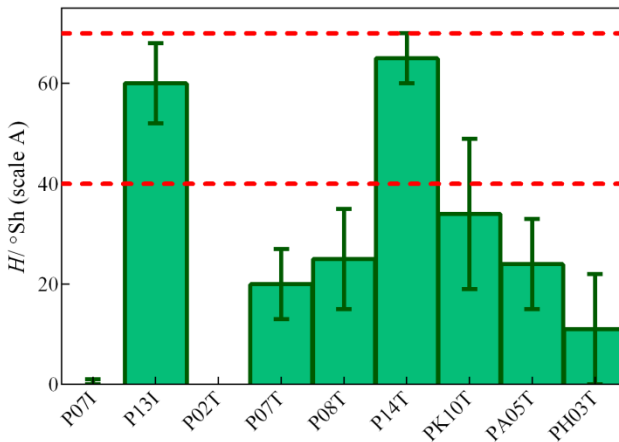


Fig. 8. Shore A hardness for propellant sample using loading method

Propellant samples with hardness values exceeding 10°Sh were measured using a helium densitometer. Based on propellant composition, the theoretical density of the mixture was calculated, taking into account additivity (ρ_{ad}) and sample porosity (P_p) in line with the formula (5):

$$P_p = \frac{\rho_{ad} - \rho_{He}}{\rho_{ad}} \cdot 100\% \quad (5)$$

Table 6 presents the obtained results. The density of heterogeneous rocket propellants, as indicated in the reference literature, is $1.73 \text{ g}\cdot\text{cm}^{-3}$ for propellants with no added aluminium, and $1.86 \text{ g}\cdot\text{cm}^{-3}$ for propellants with added aluminium [16].

The highest values of density were recorded for the PA05T and PH03T samples, and exceeded $1.73 \text{ g}\cdot\text{cm}^{-3}$. The values of density for the propellant samples with and without added aluminium were 5.8-9.2% lower than the reference literature data.

The porosity of all samples was up to 8%, which was connected with the open porosity of the granules, not filled under the formation pressure of 1 MPa.

Table 6. Density and porosity for propellant samples using the loading method

Symbol	$\rho_{\text{He}}/\text{g}\cdot\text{cm}^{-3}$	$\rho_{\text{ad}}/\text{g}\cdot\text{cm}^{-3}$	$P_p/\%$
P13I	1.6319 ± 0.0014	1.7727	7.9 ± 0.1
P07T	1.6370 ± 0.0029	1.7243	5.1 ± 0.3
P08T	1.6268 ± 0.0011	1.6975	4.2 ± 0.1
P14T	1.5886 ± 0.0015	1.6927	6.1 ± 0.1
PK10T	1.5576 ± 0.0017	1.6432	5.2 ± 0.2
PA05T	1.7600 ± 0.0021	1.8649	5.6 ± 0.2
PH03T	1.7229 ± 0.0027	1.7927	3.9 ± 0.3

To determine the thermal stability of the propellant samples, TG measurements were taken. Sample charts of the mass derivatives (temperatures for all samples) and the dependence of heat flow on temperature are presented in Figure 9. At a temperature of 246.2°C , it was possible to observe an endothermic process, which corresponded to AP changing its form from orthorhombic to cubic. The decomposition of the samples was a multi-stage process with two processes being the most evident – the first beginning above 250°C and the other at 405°C . All the tested samples had similar levels of thermal stability.

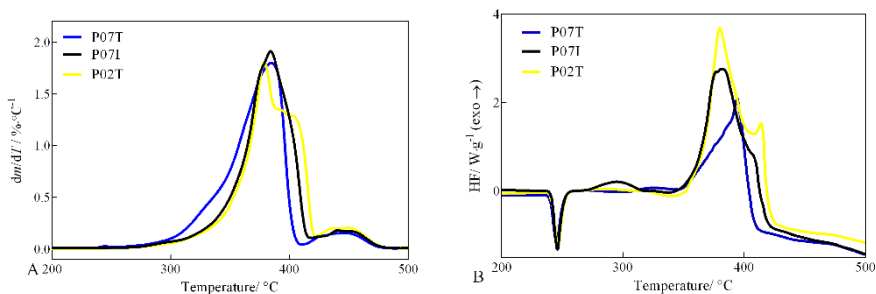


Fig. 9. A – Mass derivatives (temperature), B – dependence of heat flow on temperature for the propellant samples (P07I, P02T, P07T)

4. CONCLUSION

The proposed loading method for forming solid propellants is a safe, inexpensive and simple way of forming solid rocket propellants.

The ammonium perchlorate originating from various sources was characterised by various dimensions and bulk densities. The content of the closed pores in all samples was below 0.8%. AP CI was selected as one material for the loading method.

Each type of aluminium dust was characterised by dimensions of under 10 μm , sphericity of up to 30%, and density similar to the reference literature density. Aluminium creates agglomerates very easily. Cross-linking agents such as TDI, DDI and IPDI mixed with HTPB have sufficient pot life to be used in the loading method.

Ten samples of propellants of various compositions were obtained using the loading method. It was observed that higher viscosities of the liquid components resulted in better arrangements of the aluminium throughout the propellant volume. Several samples were characterised by hardness values of more than 40°Sh (scale A, temperature $23.0 \pm 2.0^\circ\text{C}$). The density of the obtained samples was 1.56-1.64 $\text{g}\cdot\text{cm}^{-3}$ for propellants with no aluminium and 1.72-1.76 $\text{g}\cdot\text{cm}^{-3}$ for those with aluminium. The closed pore contents in the samples was below 8%, which was related to the open porosity of the granules, remaining unfilled during a formation pressure of 1 MPa. All the samples had similar levels of thermal stability.

ACKNOWLEDGEMENT

The author would like to thank the staff of the Institute of Industrial Organic Chemistry and Mesko S.A. for the provision of samples of the materials necessary to form the rocket propellants.

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Opracowanie podstaw fizykochemicznych formowania heterogenicznych paliw raketowych metodą zasypową

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Streszczenie. Opracowano podstawy fizykochemiczne do formowania paliw metodą zasypową. Zaletami metody zasypowej jest możliwość uzyskania największej gęstości upakowania cząstek stałych, nie wymaga stosowania kosztownych urządzeń oraz nie przeprowadza się operacji mechanicznych, które stwarzają zagrożenie pożarowo-wybuchowe. Określono wymiary, gęstość helową i nasypową, zawartość porów zamkniętych oraz wolnych przestrzeni międzyziarnowych i porów otwartych, stabilność termiczną granulowanego chloranu(VII) amonu (AP) pochodzącego z różnych źródeł. Pozyskane materiały różniły się pod względem wymiarów, kulistości, gęstości nasypowej oraz zawartości porów zamkniętych oraz wolnych przestrzeni międzyziarnowych i porów otwartych. Na podstawie badań wytypowano granulaty o największej kulistości i gęstości nasypowej. Przeprowadzono pomiary wymiarów, kulistości i gęstości dla pyłu aluminiowego pochodzącego z różnych źródeł. Określono czas życia technologicznego dla układów prepolimer+środek sieciujący (diizocyjanian). Wytypowano środki sieciujące, których czasy życia technologicznego wynosiły powyżej 3,0 h w temperaturze 70°C. Na podstawie przeprowadzonych badań wytypowano materiały, które stosowano w formowaniu heterogenicznych paliw raketowych metodą zasypową. Otrzymane próbki paliw metodą zasypową scharakteryzowano pod względem parametrów fizykochemicznych.

Słowa kluczowe: technologia chemiczna, formowanie paliw, heterogeniczne paliwa, sieciowanie, metoda zasypowa