

The influence of some factors on the characteristics of metallized incendiary mixtures

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ABSTRACT

In this article, the influence of some factors on the characteristics of metallized incendiary mixtures (pyrogels) was studied. The pyrogel-type contains benzene, polyisobutylene, magnesium, aluminum-magnesium alloy, sodium nitrate, and different additives. It is shown that the molecular weight of polyisobutylene, the amount of aluminum-magnesium alloy, the particle size of metal combustibles, and the amount and type of polyester threads have different effects on the heat of combustion, flash point, burning rate, flame height, and adhesion to the material surface. The optimal pyrogel contains 38-40% thickened benzene - PIB with molecular weight 175000-225000, 25-27% aluminum-magnesium alloy with particle size $\leq 160 \mu\text{m}$, 10-12% magnesium powder with particle size $\leq 180 \mu\text{m}$, 15% sodium nitrate, 8% phenol-formaldehyde resin and 1.5% (over 100%) 3D polyester threads.

Keywords: Pyrogel; Polyisobutylene; Magnesium; Aluminum-magnesium alloy; The heat of combustion; Adhesion.

1. INTRODUCTION

Incendiary weapons are widely used for various purposes. Incendiary weapons are designed to destroy enemy manpower in shelters, buildings, and bunkers; to destroy sheltered weapons, structures, and warehouses by fires with a largely warm, high temperature and a large space of volume [1-3].

The main incendiary mixtures include napalm, thermite compositions, self-igniting incendiary mixture, and metallized mixtures (pyrogels) [3-6]. Depending on the compositions, incendiary mixtures are divided into 3 main groups: incendiary mixtures based on petroleum products, metallized mixtures, and incendiary mixtures based on thermite compositions [2, 3, 7]. Incendiary mixtures based on petroleum products (for example, napalm) have the disadvantage of being “lumpy” and having poor adhesion to the material surface. Incendiary mixtures based on thermite compositions often burn locally because of the insignificant fragmentation of the shell. In addition, a large amount of strong igniter is required to ignite these mixtures. Pyrogels, which are easy to ignite, have good adhesion to target surfaces, high combustion temperature (1200-1600 °C), long burning time, and high combustion heat.

The pyrogels may include metal powder (Al, Li, Mg, Bo,...); oxidizer - perchlorate and nitrate salts of Li, K, Al, Na; polymers and liquid hydrocarbons [8]. However, the above mixtures are difficult to burn, burn without smoke, and easily extinguished by water and sand. According to [9], the pyrogels may consist of: liquid hydrocarbons - polyisobutylene or polystyrene; metal (<30%) - Mg, Al or aluminum-magnesium alloy, NaNO_3 (<20%), powder Si, and red phosphorus. Other pyrogels contain combustible metals (Al, Mg, Na); heavy oil products (asphalt, fuel oil); some combustible polymers (isobutyl methacrylate, polybutadiene). Note that napalm-B is composed of polystyrene and used much more efficiently than conventional napalm. Napalm-B has good adhesion to various material surfaces, even wet surfaces. The pyrogels can include: liquid hydrocarbons, metals, non-metals, nitrate salts, polyisobutylene, and polyester in the form of threads [10-17]. For the American pyrogel PT-1, the total mass fraction of oil

products is 33% [13]. Natural rubber and isobutyl methacrylate are effective thickeners, but their application is more difficult than that of polyisobutylene.

It is obvious that the studies of pyrogels were carried out a long time ago. However, most of the previous studies do not present specific characteristics. The purpose of this work is to study the influence of some factors (polymer molecular weight; content and size of metal combustibles, polyester thread content) on the characteristics of pyrogels (swelling time, adhesion, combustion heat, flash point, flame height, burning time). The released combustion heat, flame height, and adhesion to material surface are the main factor of pyrogels. As a result of the study, the optimal pyrogel-type composition was chosen. The obtained pyrogel can be used in weapon RPO-Z to destroy flammable targets: trucks, canvases, temporary houses, and warehouses,... damage the energy of the opponent or create a large fire to interfere with the opponent's fighting action.

2. MATERIALS AND METHODS

2.1. Materials

The samples of the study are pyrogels based on benzene, aluminum-magnesium alloy Al_3Mg_4 , magnesium Mg, polyisobutylene rubber (PIB), oxidizer NaNO_3 , phenol-formaldehyde resin (PF), and polyester thread:

- Benzene (according to QCVN 1: 2020/BKHCHN) with an octane content $\geq 92\%$, a mass fraction of Pb ≤ 0.005 g/l, a mass fraction of heavy metals ≤ 5 mg/l.

- Aluminum-magnesium powder (PAM, Al_3Mg_4 - according to GOST 5593-78) with purity $\geq 99\%$, mass fraction of Fe $\leq 0.3\%$. Brands are: PAM-2 (particle size 140-315 μm), PAM-3 (particle size 71-160 μm); PAM-4 (particle size ≤ 80 μm).

- Powder of magnesium (Mg - according to GOST 6001-79) with purity $\geq 99\%$, mass fraction of Fe $\leq 0.05\%$. Brands are: MPF-2 (particle size 140-250 μm), MPF-3 (particle size 100-180 μm); MPF -4 (particle size ≤ 100 μm).

- PF (according to GOST 18694-80) with a mass fraction of free phenol $\leq 7\%$, a mass fraction of insoluble impurities $\leq 0.03\%$.

- PIB (according to GOST 13303-86) with a mass fraction of ash $\leq 0.3\%$;

- NaNO_3 with purity $\geq 98.5\%$, insoluble residues in water $\leq 0.15\%$.

- Polyester thread - from Viet Nam with a thickness of 3-7 Denier (D) (0.33-0.77 mg/m).

2.2. Methods

2.2.1. Scheme for the manufacture of pyrogels

The process of manufacturing samples of mixtures includes the following steps (fig. 1):

- Preparation of components (drying, grinding, preparation for sorting). Depending on the amount of drying of the raw materials and the manufacturing capacity, different types of dryers such as convection dryers, and vacuum dryers,... were used. For metal powders Mg and aluminum-magnesium, packed and stored in metal boxes, drying is not necessary. For oxidizers and additives, the moisture requirements after drying and before being put into manufacture are not exceeding 0.2%. The particle size of oxidizers and additives can be ground to apply. The particle size of Mg and aluminum-magnesium is crushed by manufacturers according to the standard for each type of metal. To ensure the density and particle size, after grinding all components need to be segmented (or checked) and remove impurities.

- Manufacturing (immersion, mixing, processing, stabilization): The mixing stage is divided into 2 stages: mix dry and wet mixing. Firstly, the mixture including metal powder, PF and oxidants is mixed on dry mixing equipment. Then, the obtained mixture is put in a closed mixing

device containing the solution obtained after the concentration of benzene and PIB.

- Stuffing, loading into battle (measuring, pumping, or formatting by compression, molding, stuffing).

Because the manufacturing process contains flammable and volatile substances (benzene), so to ensure safety, it is necessary to use a mixing device with high tightness, plus the filling process must be vacuumed. To ensure continuity the whole stirring and filling process should be done in one unit. Note that:

- The mixer for mixing dry particles with an inclined shaft with a capacity of 15 liters consists of a thermostatic shell, inside the mixing chamber, and rods run along the mixing tank.

- A closed system of a mixing apparatus (fig. 2) with a closed tank with a capacity of 25 liters (fig. 3), in which different types of agitators can be changed, is used for thickening benzene, closed mixing, and stabilization of pyrogels.

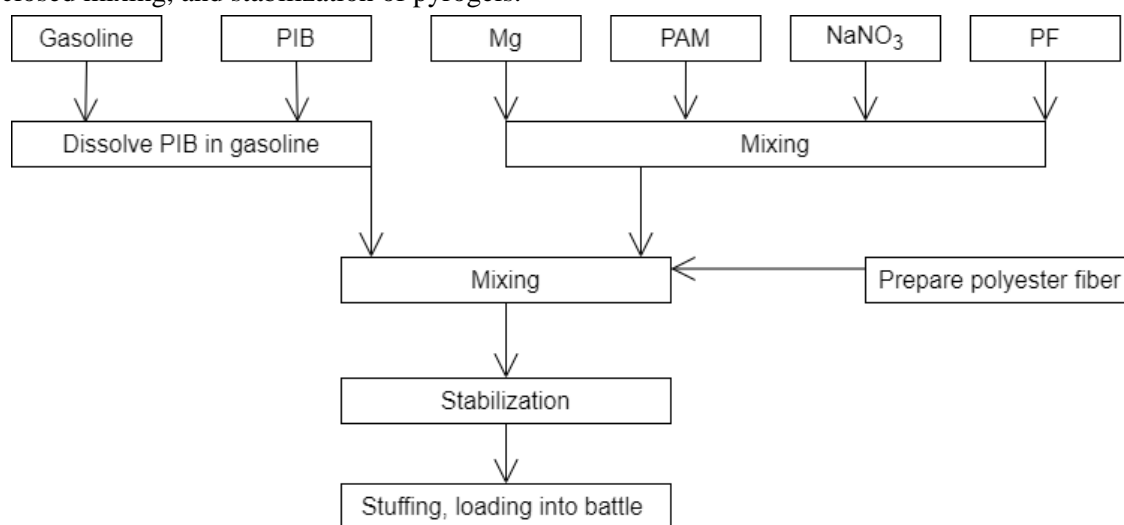


Figure 1. Scheme for the manufacture of pyrogels.

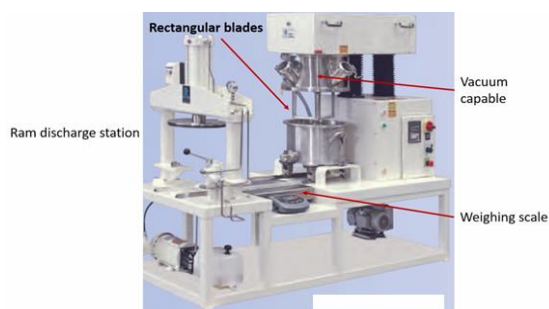


Figure 2. Closed mixer system.



Figure 3. Closed tank.

2.2.2. Methods for determining parameters

The experiments were conducted at the laboratory of the School of Special Machine Engineering - Le Quy Don Technical University, and the Institute of propellants and Explosive - General Department of Military Industries and Manufacture.

The viscosity of the polymer solution is measured by viscometer Brookfield.

The heat of combustion (Q) is determined according to TCVN/QS 889:2019 on a PARR 6100 with heat bomb 1108. The heat bomb is charged with oxygen for 1 minute before placing the

bucket. The amount of oxygen up to 30 atm is loaded into the test bomb. Determination accuracy was $\pm 5\%$. The final result is the average of the two results.

The flash point (T_f) is determined on the device T900, with operating temperature from 200 to 950 $^{\circ}\text{C}$, with an error of $\pm 5^{\circ}\text{C}$. Flash time ≤ 3 s.

Determining the burning rate (r_b) and flame height (h_f) was performed by using the FASTCAM SA5 high-speed camera. The sample weight is 50 g. The sample is prepared in the $\Phi 50$ cylinder, and the density of the sample reaches 1.05 g/cm^3 (as the density of the incendiary mixture in a grenade).

The adhesion of the material (a_m) was determined with an $\Phi 50$ adhesion meter disk made of steel with a surface roughness of $R_a = 10\text{-}20$; the density of the sample on the disk reaches 1.4 g/cm^3 .

The combustion temperature is determined by tungsten rhenium thermocouple wire on the burning surface of the sample. The combustion temperature was measured for a sample with a density of 1.05 g/cm^3 , a diameter - 12 mm and a height - 24 mm. Determination accuracy was $\pm 5\%$.

3. RESULTS AND DISCUSSION

3.1. Thermodynamic calculations of compositions

In this work, thermodynamic calculations were carried out using the maximum entropy method. The total mass fraction of oil products and polymer in Russian pyrogels intended for equipping incendiary ammunition is 38.4% and 3.6%, respectively [14]. So, the selected calculated pyrogels contain 30-40% benzene, 3.5% PIB, 7.5-8.5% PF, 15.5–18% oxidizer, and PAM with Mg (table 1).

Table 1. Composition and properties of mixtures of the pyrogel type.

Component	Specific heat of combustion ΔH_C^0 (kcal/kg)	Mass fraction, %					
		M1	M2	M3	M4	M5	M6
Benzene	-10717	30	32	34	36	38	40
Al_3Mg_4	-6598	29	28	27	26	25	24
Mg	- 5909	11	10,5	10	10	10	9
NaNO_3	+712	18	18	17	17	16	15.5
PF	-744.8	8.5	8	8.5	7.5	7.5	8
PIB	-476.5	3.5					
Oxygen balance K_b , %		-158	-163	-170	-174	-180	-186
Q, kcal/kg		5730	5845	5975	6116	6271	6368
Combustion products in the form of solid (to oxides: Al_2O_3 , MgO , Na_2O), $\text{g}/\text{g}_{\text{mixture}}$		0.83	0.74	0.71	0.69	0.67	0.63

It has been shown that the oxygen balance of this pyrogel-type is rather negative. The calculation of the heat of combustion, considered under conditions of complete combustion in the air according to the specific heat of combustion (ΔH_C^0) of each component, shows that all pyrogels have a high heat of combustion [17-23].

With an increase in the mass fraction of benzene in the mixture, the heat of combustion increases (this is natural since the specific heat of combustion of benzene is the highest in the

mixture). It can be seen from the calculation results that samples M5 and M6 (with 38 and 40% benzene) have the highest calorific value. It should be noted that for pyrogels, in addition to a high calorific value, a high temperature and a high concentration of condensed solid slag in the combustion product are also required, which means that the content of metals and alloy should be sufficiently high.

3.2. Influence of PIB molecular weight on swelling, solubility in benzene, and adhesion to the material surface

Depending on its molecular weight (M), the PIB has a different ability to swell and dissolve it in benzene. To study the swelling and solubility of PIB with different molecular weight in benzene, PIB was cut into small pieces (with a length of each side no more than 5 mm) and added to benzene with a mass ratio of benzene / PIB ~ 10/1, with a mixing frequency of 2-3 h/time (under the condition of stirring for 5 minutes at a speed of 100 rpm, temperature 25 °C). The sample M5 was chosen for this study. The results are given in table 2.

Table 2. Influence of PIB molecular weight on pyrogel characteristics.

Property	P-200	P-155	P-85	P-20 (liquid)
M	175000-225000	135000-175000	70000-100000	15000-25000
Swelling and dissolution time in benzene*	about 30 h.	about 20 h.	less 16 h.	-
Viscosity, Pa.s	15000	3500	150	-
Mix condition	Homogeneous paste	Homogeneous paste	liquid solution	liquid solution
Stability after mixing with solid components (by the sample M5)	The resulting mixture well covers the solids without forming a "lumpy"	The re"ulting mixture well covers the solids without forming a "lumpy"	Stability with solids is not good, solids settle	-
$a_m, \text{g/cm}^2$	23.1	21.4	< 17.3	-
Adhesiveness of pyrogels with polyester threads	good	good	Settling during storage	-

* The time of swelling and dissolution is calculated from the time the polymer is put into the solvent until the polymer swells and dissolves completely (no air bubbles, phase zones in the polymer solution).

The solution of benzene with PIB brand P-20 (M = 15000-25000), which is in a liquid state does not adhere to solid particles of the components. For PIB brand P-85 (M = 70000-100000), the accumulation time in benzene is less than 16 hours, however, the resulting mixture has a low viscosity, which leads to precipitation when used with solid components. The swelling and dissolution time of PIB of brands P-155 and P-200 (about 20 hours and 30 hours) in benzene is longer than PIB with low molecular weights, and the mixture enters the form of a paste, a good coating of solid components, without clumping when combined. Pyrogels with P-155 and P-200

have more adhesion to the surface of the material $21.4 \div 23.1 \text{ g/cm}^2$. The PIB brand P200 was selected for the next studies.

3.3. Influence of Al_3Mg_4 -alloy content on pyrogel characteristics

The main combustible substances in pyrogels include petroleum products (benzene and their derivatives) and metal powders. Oil products make a large amount in the pyrogels and generate large combustion heat withstanding the burning time to ensure heat transfer to the target, and also transferring the heat of ignition to the rest of the mixture. Combustion of benzene mainly occurs in the gas phase. The metal powders added to pyrogels create combustion intensity and high temperature, and molten slag are formed. Combustion of powders of metals and alloys occurs after receiving heat from the combustion of benzene. The use of PAM alloy helps increase the combustion rate because of its chemical stability compared to using only powders Al and Mg (in a humid environment, PAM alloys do not react with nitrate salts). The presence of Mg powder helps to reduce the ignition temperature of the mixture of solid particles.

Thus, a change in the content of combustible metals leads to a change in the heat of combustion, as well as the burning rate. The influence of the content of PAM on the calorific value, burning rate, flame height, and adhesion to the surface of the material for pyrogel with a mass fraction of Mg - 10%, NaNO_3 - 15%, PF - 8%, polyester threads - 1.5% (- over 100%) – was studied.

Table 3. Influence of Al_3Mg_4 -alloy content on pyrogel characteristics.

Mass fraction with PIB, %	Mass fraction Al_3Mg_4 , %	Q, kcal/kg	T_f , °C	r_b , mm/s	h_f , m	a_m , g/cm^2
35	32	5380	610	3.9	0.9-1.3	18.9
38	29	5811	595	3.0	0.8-1.1	21.2
41	26	6556	585	2.5	0.8-1.1	21.6
44	23	6735	580	1.9	0.5-0.9	23.4
47	20	-	580	1.6	0.3-0.7	-

Table 3 shows that with an increase in the content of benzene with PIB (i.e., with a decrease in the content of Al_3Mg_4), the calorific value increases, the flash point decreases, and the burning rate and flame height decrease. The samples with a high mass fraction of the alloy burn with good intensity, creating a large volume of fires and dissipating a more intense amount of metal slag; however, adhesion to the material surface is reduced. For example, for the sample with 32% Al_3Mg_4 adhesion is 18.9 g/cm^3 , and for the sample with 23% alloy, it is 23.4 g/cm^2 . The combustion of pyrogels occurs due to the diffusion of benzene and air vapors into the fire zone. The combustion process spreads over the surface of the pyrogel. Thus, after a fire occurs, the next combustion process is the self-sustaining combustion of benzene and metal powder. The burning rate of benzene is lower than that of Mg powder and Al_3Mg_4 alloy. By increasing the content of benzene with PIB, the level of coating to the surface of the solid particles increases, which leads to a decrease in the intensity of the fire.

The flash point is the temperature, at which the mixture of benzene vapor and can ignite in air. So, with a higher content of benzene in the composition, diffusion to the gas phase to reach the flash point will be lower. For example, the flash point of a sample with 47% benzene with PIB is 580 K, and for a sample with 35% - 610 K. So, the difference between the flash points of the given samples is small. In this case, the pyrogel with ~26% Al_3Mg_4 is the best choice for manufacturing pyrogels.

3.4. Influence of particle size of Mg and Al₃Mg₄ alloy on pyrogel characteristics

The samples for research were benzene with PIB - 40%, Al₃Mg₄ alloy - 25%, magnesium powder - 12%, NaNO₃ - 15%, PF - 8%, and polyester threads - 1.5% (over 100%). The results are shown in table 4.

Table 4. Influence of particle size of Mg and Al₃Mg₄ alloy on pyrogel characteristics.

particle size of Al ₃ Mg ₄ , μm	particle size of Mg, μm	particle size of NaNO ₃ , μm	particle size of PF, μm	r _b , mm/s	h _f , m	T _f , °C	a _m , g/cm ²
140-315 (PAM-2)	140-250 (MPF-2)	≤ 100	≤ 100	1.9	0.5÷0.9	610	19.5
71-160 (PAM-3)	100-180 (MPF-3)	≤ 50	≤ 100	2.7	0.8÷1.1	580	21.6
≤ 80 (PAM-4)	≤ 100 (MPF-4)	≤ 50	≤ 100	3.2	0.8÷1.1	580	22.4

Table 4 shows that the sample with larger metal particles burns at a lower burning rate and has lower flame height. The sample with PAM-2 and MPF-2 has the smallest flame (height of 0.5-0.9 m), and the sample with PAM-4 and MPF-4 has the highest flame (height of 0.8-1.1 m). When the particle size of metals and alloys is smaller, the similarity with the particle size of NaNO₃ and PF is good, and the total surface area of the particles is larger, so the burning rate is higher and the burning is more intense. For the sample with PAM-2 and MPF-2, they burn several times slower than for other samples. In addition, adhesion to the surface of the material is increased when the particle size of the components is smaller due to better coverage to the surface of the solid particles. The flash point does not change much - from 580 to 610 K.

It should be noted that the sample with PAM-2 and MPF-2 has a discrete condition and is not in a pasty state. The resulting mixture after mixing the solid particles is uneven, the components settle, and the stability and adhesion of the pyrogel to polyester threads are poor. On the contrary, for the other two samples, the stability and adhesion of the pyrogel to polyester threads are good. So, PAM-3 and PAM-4 can be chosen for the production of pyrogels.

3.5. Influence of polyester threads on the pyrogel characteristics

Polyester threads are introduced into the mixture in order to create branched systems of pores in the pyrogel during combustion, thereby facilitating the introduction of gaseous decomposition products of oxidizing and non-oxidizing substances into the combustible mixture and solving the problems of component deposition during long-term development and storage. Then the pyrogel retains its state during long-term storage [16]. In addition, the use of polyester fibers eliminates the separation of combustibles when they hit the target, contributing to the concentration of the heat source during combustion.

Polyester threads with a thickness of 3 Denier (D) and 7D were investigated. Polyester bundles were dried and loosened together with the components in the process of mixing dry particles, then they are mixed in a closed system of the mixing apparatus. Mass fraction of components as in M5. The results are shown in table 5.

With an increase in the content of polyester threads, the burning rate increases. For the sample with a larger number of threads, a more branched pore system is created, and possibly high degrees of conversion of organic fuels are provided, so the combustion intensity is stronger. However, when the content of polyester threads increases, the density of the pyrogel decreases,

and the adhesion to the surface of the material decreases. For the samples with 7D fiber, manufacturing efficiency is reduced. The pyrogel does not have a pasty state and its adhesion to the surface of the material is low. And so, for the development of effective pyrogels, the polyester threads 3D with a share of 1.5% (over 100%) should be chosen for use.

Table 5. Influence of polyester threads on the pyrogel characteristics.

Mass fraction of polyester threads (over 100%), %	Mass of pieces of the mixture, g	Manufacturing efficiency **, %	r_b , mm/s	h_f , m	a_m , g/cm ²
0,5 (3D)	48 – 75	51	1.5	0.3 – 0.5	23.0
1,5 (3D)	95 – 136	92	2.7	0.8 – 1.1	21.6
2,5 (3D)	86 – 120	90	3.1	0.8 – 1.0	18.7
1,5 (7D)	72 – 89	63	3.1	0.9 - 1.1	16.7
2,5 (7D)	71 – 96	71	3.9	0.9 - 1.2	15.2

** The ratio of the mass ratio of the mixture attached to the polyester threads/total mass

4. CONCLUSIONS

In this article, the influence of some factors on the characteristics of incendiary mixtures of the pyrogel type was investigated. It is shown that the pyrogels have a low oxygen balance and a high calorific value. PIB swells in benzene (weight ratio PIB/benzene = 1/10) with long mixing and storage times. With a decrease in the amount of Al₄Mg₃ alloy, the flash point and burning rate of the samples decrease, while the combustion heat and adhesion increase. The samples with smaller particle sizes of metal particles have better properties than the samples with larger ones. Polyester threads are used to create a mesh and avoid component delamination. Pyrogel, containing (38-40)% thickened benzene (PIB with molecular weight M=175000-225000), (25-27)% PAM (particle size ≤ 160 μm), (10-12)% magnesium powder (particle size ≤ 180 μm), 15% NaNO₃, 8% PF and 1.5% (over 100%) 3D polyester threads, is the optimal incendiary metallized mixture. This pyrogel burns well and its experimental combustion temperature can reach 1800 K.

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TÓM TẮT

Nghiên cứu ảnh hưởng của một số yếu tố đến đặc trưng của hỗn hợp cháy chứa kim loại

Trong bài báo này, ảnh hưởng của một số yếu tố đến đặc tính của hỗn hợp cháy chứa kim loại (pyrogels) đã được nghiên cứu. Các hỗn hợp pyrogel thường chứa benzen, polyisobutylene, magiê, hợp kim nhôm-magiê, natri nitrat và các chất phụ gia khác nhau. Kết quả chỉ ra rằng, khối lượng phân tử của polyisobutylene, hàm lượng hợp kim nhôm-magiê, kích thước hạt của chất cháy kim loại, hàm lượng và loại sợi polyester có ảnh hưởng khác nhau đến nhiệt lượng cháy, điểm chớp cháy, tốc độ cháy, chiều cao ngọn lửa, và độ bám dính vào bề mặt vật liệu của hỗn hợp thuốc pyrogel. Qua nghiên cứu, pyrogel tối ưu chứa 38-40% benzen được làm đặc bởi PIB với khối lượng phân tử 175000-225000, 25-27% hợp kim nhôm-magiê với kích thước hạt $\leq 160 \mu\text{m}$, 10-12% bột magiê với kích thước hạt $\leq 180 \mu\text{m}$, 15% natri nitrat, 8% nhựa phenol-formaldehyde và 1,5% (thêm ngoài 100%) sợi polyester 3D.

Từ khóa: Pyrogel; Xăng; Magiê; Hợp kim nhôm-magiê; Nhiệt lượng cháy; Độ bám dính.