INFLUENCE OF TECHNOLOGICAL PROCESS ON THERMAL AND MECHANICAL CHARACTERISTICS OF PYROTECHNIC COMPOSITIONS BASED ON RED PHOSPHORUS

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Abstract

This study studied the technological process of pyrotechnic compositions based on red phosphorus (RP). In addition, the influence of the RP mixing method on thermal and mechanical characteristics was also investigated. Measurements such as scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) are used to evaluate the completeness of samples; Burning rate, burning temperature, and thermogravimetry/ differential thermal analysis (TG/DTA) are used to assess thermal characteristics; Compressive strength is used to determine compressive strength, in addition, ignition temperature and friction sensitivity are also used to evaluate the safety of samples. The results showed that the samples had good completion, and the compatibility of the ingredients in the pyrotechnic composition was high. Samples with RP mixed after granulation have high friction and temperature sensitivity but have better compressive strength than samples with 100% RP mixed before granulation. The samples have similar mass burning rates in the range of 0.035 - 0.063 $g.cm^{-1}$, and the burning temperature of these samples is in the range of 1100 - 1500^oC.

Keywords: *Technological process; pyrotechnics; red phosphorus (RP).*

1. Introduction

Pyrotechnic composition based on the RP is commonly used for various special applications, such as ground signal transmission, signals, and maritime special operations [1]; white smoke is also used to hide targets [2]. Smoke-generated obscuration systems provide instant invisibility. This smoke screen is formed between opposing personel and equipment, temporarily blocking electro-optical sensors, which can significantly increase the survivability of humans in special situations. In special operations, in addition to light detection and ranging (LiDAR) [3] and radio detection and ranging (RADAR) [4] technologies, the most common observation technologies are often passive sensors based on image enhancement and thermal observation modes [5].

Pyrotechnic smoke based on the RP has been studied for a long time [6-8], and until now, this is still an attractive subject of research by scientists worldwide [9-11]. Infrared Ray smoke generators are still produced in the special, mainly based on the RP [12]. Most

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of the above works study the influence of components on covering characteristics (including infrared emission and laser interference). However, there are still very few publications researching the manufacturing technology and its impact on the burning characteristics of the compositions.

The technological process of making pyrotechnics in general and pyrotechnic smoke based on the RP, in particular, is according to the regulations of dry mixing of the combustible agents and oxidizer separately, then wet mixing of the two ingredients in proportion to the solvent and proceeding with granulation. However, by studying some energy characteristics, smoke coverage, and infrared emission, the author determined that mixing the RP into composition can affect pyrotechnic compositions' energy and mechanical characteristics.

This study studies the technological process of making composition based on the RP with a change in the second RP injection rate into the mixture, gradually increasing from 0% to 50% (step of 10%) by weight. These samples were evaluated for completeness through scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDX), and optical microscope (OM). Burning rate measurements and TG/DTA analysis were also used to measure the energy characteristics of pyrotechnic compositions. In addition, friction sensitivity, ignition temperature, and mechanical strength were also investigated.

2. Materials and methods

2.1. Materials

2.1.1. Tested materials

Table 1. The parameters of the reagents

Reagent	Formula	Source	Size, purity	% by weight
Red phosphorus	P	China	42 ÷ 62 µm, purity ≥ 99%	63.5
Sodium nitrate	$NaNO_3$	Z Co. Ltd., Vietnam	≤ 63 µm, purity ≥ 98%	7.0
Aluminum-magnesium alloys	AlMg		147 ÷ 162 µm, purity ≥ 98%	15.0
Viton	$C_{10}H_7F_{13}$	Xilong Co. Ltd., China	purity ≥ 99%	8.0
PTFE	$(C_2F_4)_n$		≤ 10 µm, purity ≥ 98%	6.0
Manganese dioxide	MnO_2		≤ 63 µm, purity ≥ 85%	0.5
Acetone	C_3H_6O		AR, purity 99.5%	-

Table 1 summarizes the purities of particle sizes and manufacturers of the reagents, along with the weight percentages of the reagents employed in pyrotechnic compositions.

2.1.2. Safety note

The RP is extremely sensitive to friction and static electricity [8, 13]. The RP and the chemicals used in the article have a high fire risk, so personal protective equipment such as face shields, gloves, and protective clothing must always be worn. Pyrotechnic products are ultimately susceptible to friction, static electricity, and impact, so all manipulations with such products must be extremely careful.

2.2. Methods

2.2.1. Preparation of the pyrotechnic compositions

A blank sample in this study was a composition of Al-Mg/PTFE/$NaNO_3$/MnO_2/Viton (at 15%/6%/7%/0.5%/8% by weight). The samples were prepared by adding a fixed amount of RP, which is 63.5% by weight, but divided into P1 and P2 with increasing P2 from 0% to 50% (step of 10% by weight). The preparation process and schematic diagram of the samples are shown in Fig. 1.

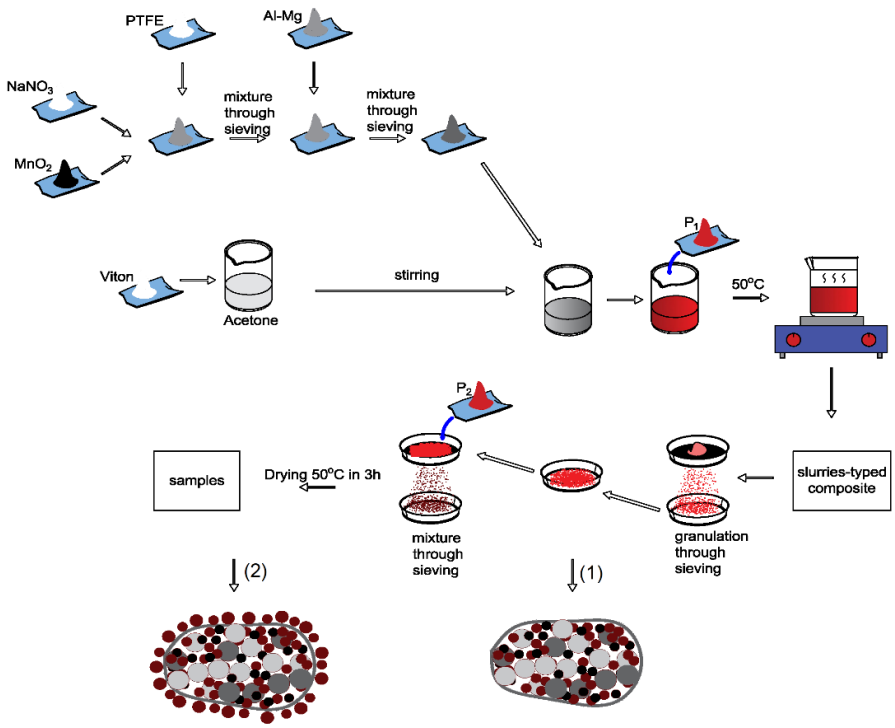

Fig. 1. The preparation process of samples.

Sample M1 was prepared by mixing 100% of the mass of the RP into the blank; in other words, 100% of the mass of the RP was mixed into the mixture before

granulation. After granulation, the samples are dried and tested. Samples M2 ÷ M6 were made by dividing the amount of the RP into two parts: P1 and P2. P1 of the RP will be mixed in before granulating, and P2 of RP will be mixed in with the pyrotechnic granules created in the above step. P1 of RP will gradually decrease from 90% to 50%, or P2 will increase from 10% to 50%, corresponding to samples named M2 to M6. Thus, with sample M1, the RP particles will be located together with other combustible agents and oxidizers in the binder (Fig. 1-(1)). Samples from M2 to M6 will have a small amount of free RP lying outside and attached to the surface of the pyrotechnic particles (Fig. 1-(2)).

2.2.2. Test methods

The scanning electron microscope (SEM) micrographs were observed with JSM-7800F scanning electron microscopy. Samples were metal sprayed in advance. The energy-dispersive X-ray spectroscopy (EDX) was tested by X-max 80. Pictures of samples were taken with a NIKON YS100 optical microscope (OM) at magnifications of ×100.

Fourier transform infrared (FTIR) spectra were recorded on the Perkin-Elmer Spectrum 400 spectrometer. The wavenumber range was set from 400 to 4000 $cm^{-1,}$ and the resolution of the spectra was 4 cm^{-1}.

The cylindrical pellet was loaded into a stainless steel tube and ignited using an electrical heating wire module as the ignition system. The entire ignition, combustion, and flame emission process was captured using a digital video camera, while a temperature measurement system monitored the flame's temperature. The digital video camera had a frame rate of 30 frames per second. The temperature measurement system consisted of a type K thermocouple with a wire diameter of 0.127 mm, which had excellent dynamic response capabilities. This thermocouple was positioned at the entrance of the stainless steel tube to record the flame temperature. A signal processor and a PC-based data acquisition system accompanied it.

To study the chemical compatibility of prepared pyrotechnic compositions, three samples, including the blank, the pure RP, and their mixture in a 50:50 mass ratio, were prepared, and their chemical compatibility was assessed using TG/DTA analysis. The test sample was positioned within an aluminum crucible, and all experiments were conducted in a nitrogen atmosphere with a heating rate of $10°C.min^{-1}$. Sample weights ranging from 3 to 5 mg were employed to measure the exothermic peak temperature and mass loss during the chemical reaction.

The experimental determination of the ignition temperature was conducted using the DT-400 test apparatus. Three samples weighing 0.2 g per one were dried, ground to an

appropriate particle size, and placed in three vertically positioned test tubes within a heating block. The temperature was systematically raised at 20°C.min^{-1} until the sample underwent deflagration. The digital control unit monitored the ignition temperature, and the average temperature from the three samples was subsequently calculated.

The BAM friction tester was employed to assess the friction sensitivity of the samples. The evaluation of friction sensitivity followed the 1 of 6 tests, defined as the minimum load required to elicit an audible or visible reaction in at least one out of six trials - the measurement range for the friction load extended from 0.5 to 360 N.

3. Results and discussions

3.1. Determine the uniformity of the pyrotechnic compositions

The pyrotechnic samples were examined by visual inspection and microscopic imaging. Images of samples under a microscope with x100 magnification are shown in Fig. 2. Figure 2a is an image of a blank sample that does not contain RP, so it is clear that the material particles are surrounded by Viton. Figure 2b and 2c are images of samples of M1 and M6, it can be seen that the red particles are RP and it is impossible to distinguish difference between these two samples at x100 magnification.

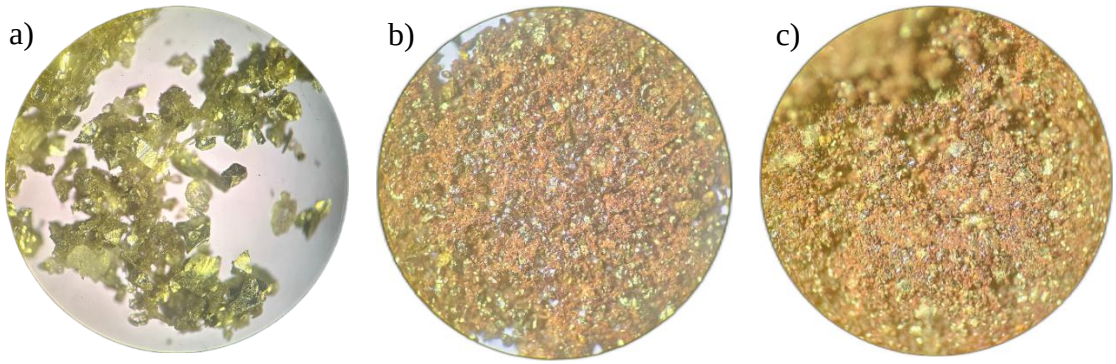

Fig. 2. Image of samples under a microscope at x100 magnification: a) the blank; b) the sample with P1/P2 ratio is 100/0; c) the sample with P1/P2 ratio is 50/50.

To check the uniformity of ingredients in the pyrotechnic compositions, the samples with P1/P2 ratio composition of 100/0 and 50/50 were investigated by SEM and EDX. SEM images of samples are shown in Fig. 3. EDX measurement results of samples are shown in Fig. 4.

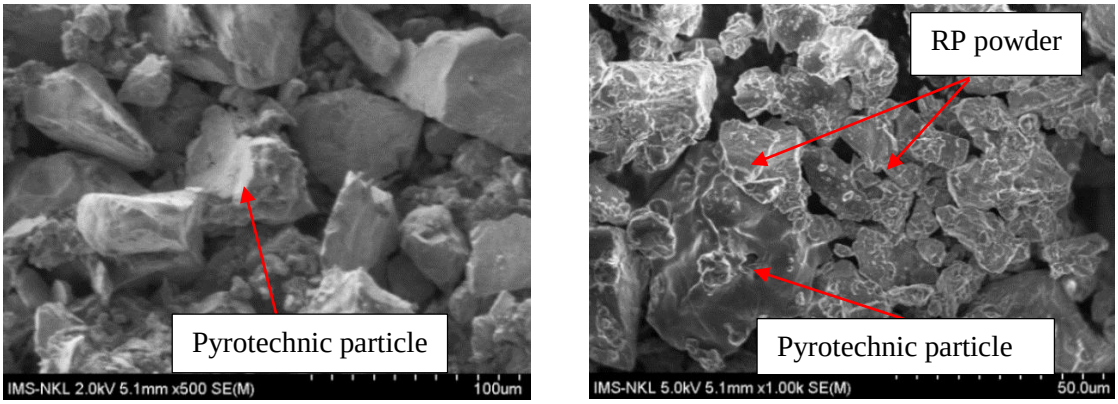

Fig. 3. SEM of samples: a) P1/P2 at ratio of 100/0; b) P1/P2 at ratio of 50/50.

Observing the images of samples in Fig. 2 and Fig. 3, it can be seen that for samples with a P1/P2 ratio of 100/0, the majority of RP particles are located in a uniform area consisting of components of pyrotechnics (metal particles, salt...) (Fig. 3a). For the sample with a P1/P2 ratio of 50/50, the RP particles adhere evenly to the entire surface, making it difficult to detect the other components (Fig. 3b).

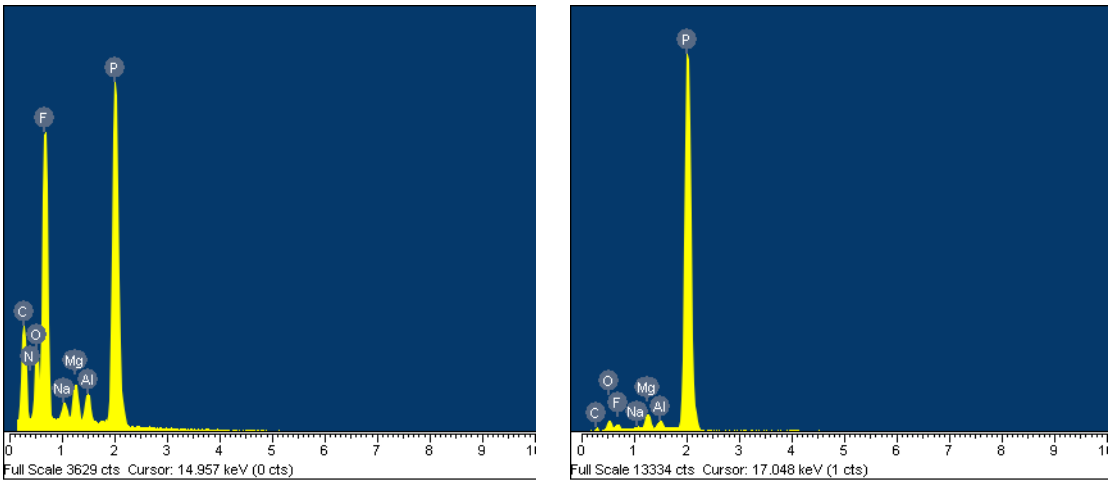

Fig. 4. EDX of samples: a) P1/P2 at ratio of 100/0; b) P1/P2 at ratio of 50/50.

It can be seen that the perfection level of the pyrotechnic compositions is quite good, and the distribution of the ingredients is relatively even. EDX measurement results show that all sample locations contain the same elemental composition. Fig. 4 shows the difference in pyrotechnic composition when the P1/P2 ratio is 50/50; almost all particles are covered by RP. That explains why the element RP appears in the EDX spectrum of the 50/50 sample almost continuously, while the peaks of other components are shallow (Fig. 4b).

3.2. The burning performance

The samples' linear burning and mass burning rates were tested to evaluate their ability to burn under atmospheric pressure conditions.

The linear burning rate of pyrotechnic composition demonstrates the law of burning and the influence of factors on the burning rate. The linear burning rate of the samples was investigated; however, for samples from M2 to M6, RP powder covers the outside of the pyrotechnic particles, so the burning time of these samples is prolonged. This causes these samples to burn incompletely. For sample M1, the RP particles are enclosed inside with other oxidizers and combustible agents; the burning ability is much better, and the burning rate is stable. The sample was stuffed into a steel tube with a diameter of 12 mm, a tube height of 25 mm, and a compression density of 1.25 $g.cm^{-3}$. The experimental results of the linear burning rate of sample M1 is 0.51 $mm.s^{-1}$. The burning image is shown in Fig. 5.

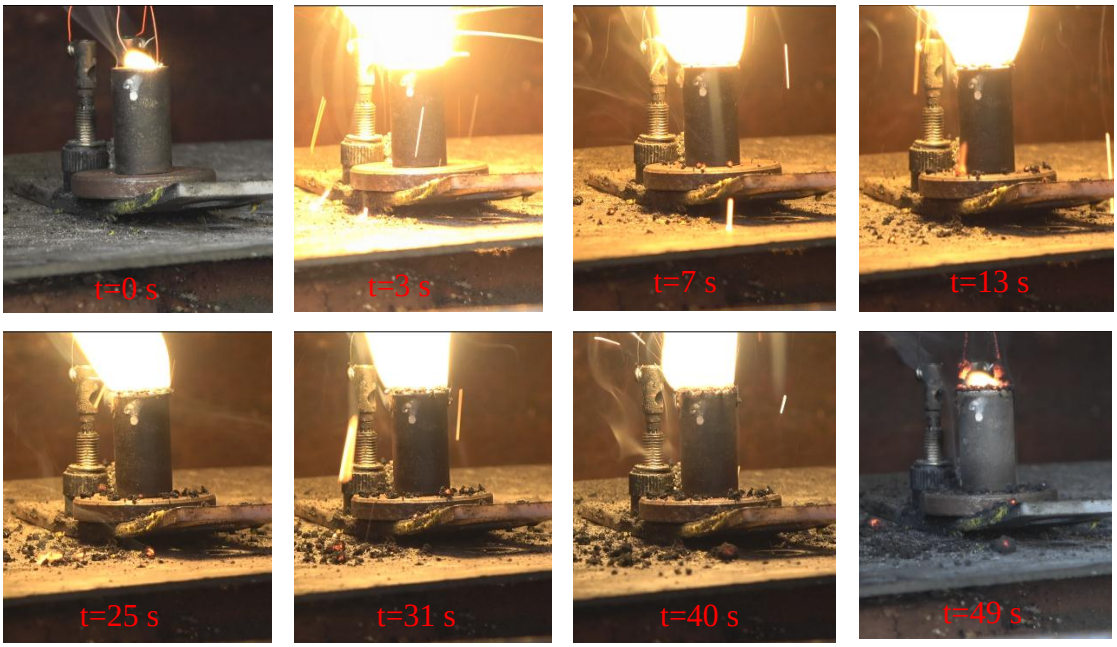

Fig. 5. A sequence of consecutive images capturing the combustion phenomenon, recorded using a digital video camera for sample M1.

The combustion of pyrotechnic composition based on RP has a very bright flame and tends to shoot small particles into the surrounding environment.

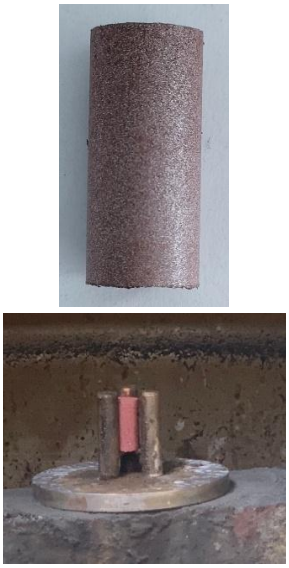

Fig. 6. RP smoke pellet (a) and mass burning rate measuring device (b).

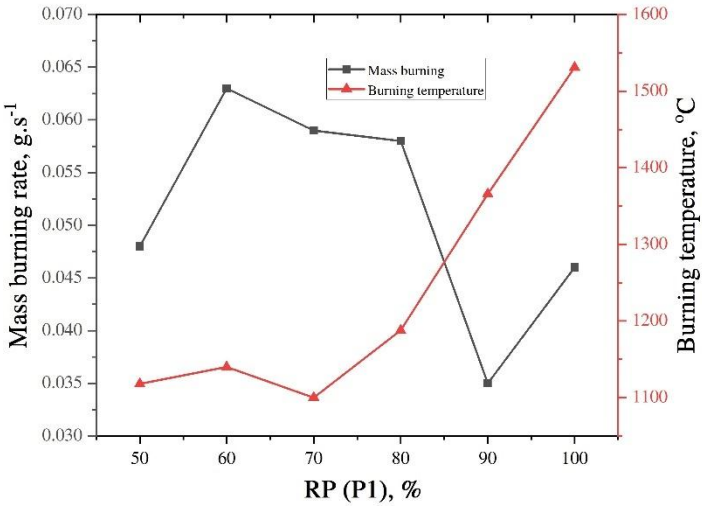

Fig. 7. The mass burning rate and burning temperature of RP compositions.

The mass burning rate of samples M1 ÷ M6 was investigated. The samples were compressed into tubes with a diameter of 12 mm, a height of 30 mm, and a density of 1.3 $g.cm^{-3,}$ as shown in Fig. 6. The measuring results of the mass burning rate of the samples are shown in Fig. 7. Besides, the temperature combustion of these samples was also studied, and shown in Fig. 7.

It can be seen that the mass burning rate of the samples is relatively uniform, does not have a clear specific trend, and is within the range of 0.035 ÷ 0.063 $g.s^{-1}$. This can be explained because the mass burning rate is often greatly influenced by external factors such as wind direction and the amount of oxygen in the air... so the mass burning rate has no particular trend as long as the samples do not vary too much in composition. In contrast, the burning temperature of the samples fluctuated between 1100°C ÷ 1500°C and tended to decrease when mixing RP outside. The burning temperature of the samples dropped with an increasing amount of free RP on the outside of the pyrotechnic particles. The reaction between RP and O_2 in the air will form P_2O_5, and the sublimation of P_2O_5 is an endothermic reaction so that it will reduce the combustion temperature.

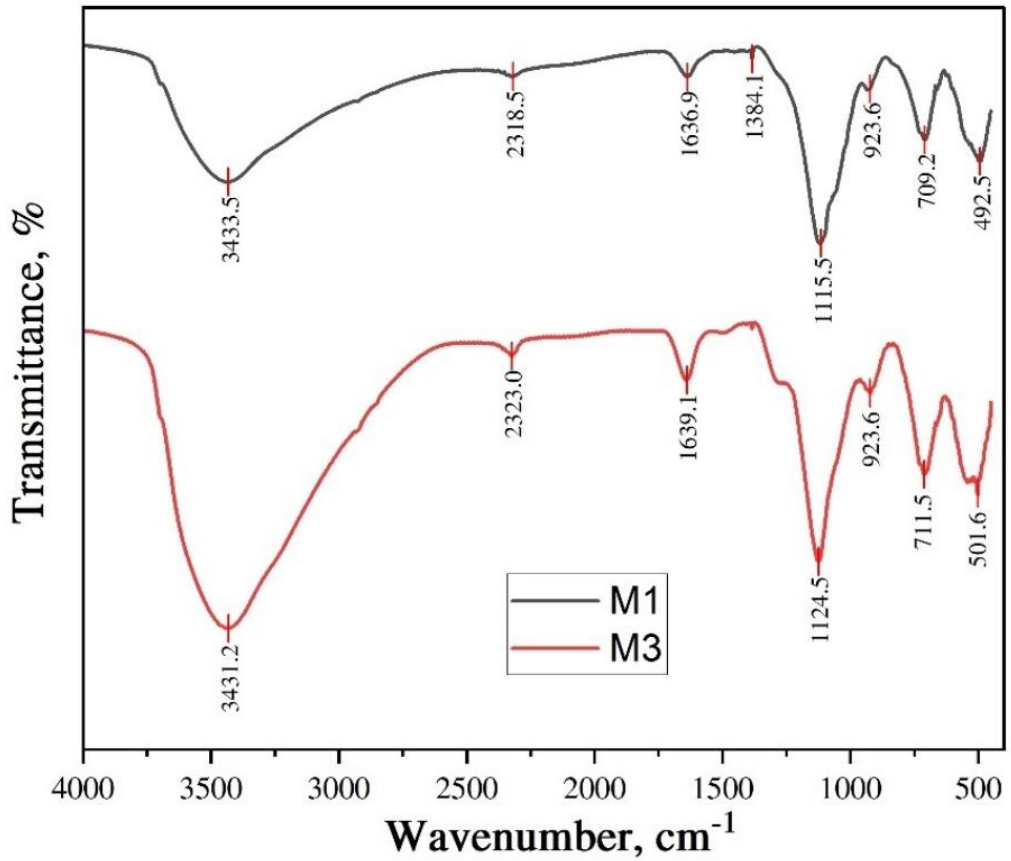

Fig. 8. The infrared spectrum of the residues of the samples M1 and M3.

Measure the IR spectrum of the solid products obtained after burned samples M1 and M3 to determine the composition of the solid phase in the combustion products. The results are shown in Fig. 8. It can be seen that the solid products of the two samples are the same. The peaks at ~3433 cm^{-1} and 1636 cm^{-1} represent the existence of H_2O. The peaks at 1384 cm^{-1} and 709 cm^{-1} are characteristic of MgF_2, while those at 492 cm^{-1} are characteristic of MgO. The peak at 1115 cm^{-1} is characteristic of P_2O_5.

3.3. Thermal analysis

To study thermal stability and compatibility using heat curves according to STANAG 4147 standards [14], Sample M0 was made with a mass ratio of 50:50 of the RP and blank following the same process as sample M1. The DTA and TGA curves of 3 samples (a) the RP, (b) the M0 with RP: blank mixture = 50:50, and (c) the blank are shown in Fig. 9 and Fig. 10.

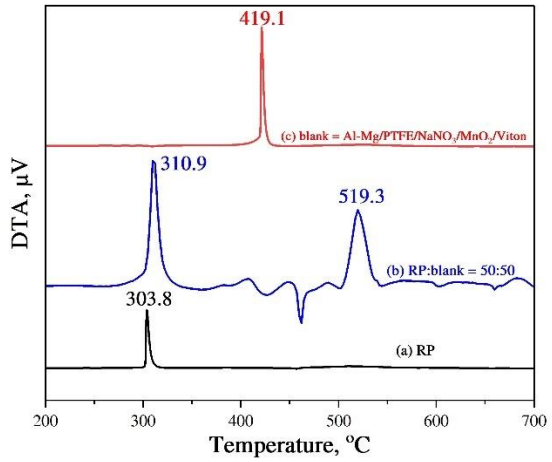

Fig. 9. The DTA curves of (a) RP, (b) RP/blank = 50:50, (c) blank (heating rate 10°C.min^{-1}; argon atmosphere).

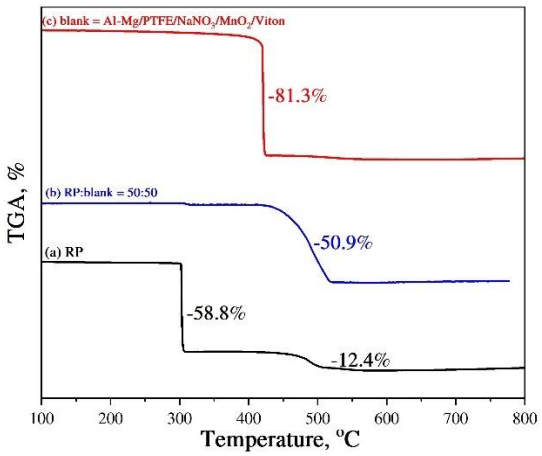

Fig. 10. The TG curves of (a) RP, (b) RP/blank = 50:50, (c) blank (heating rate 10°C.min^{-1}; argon atmosphere).

It can be seen that sample M0 (curves b) has two clear decomposition peaks with corresponding peak temperatures of 310.9°C and 519.3°C. The peak at 310.9°C may be in concert with the reaction of RP with oxygen from the decomposed oxidizer-$NaNO_3$. Because the reaction occurs in an inert gas environment, it happens slowly and for a long time; during this period, the mass of the sample almost does not change (Fig. 10 - line b). At a temperature of about 500°C, the sample's combustion reaction occurs violently, accompanied by a weight loss of more than 50% (Fig. 10 - line b) - the residue for nearly 50% of the total mass of the sample. The DTA curves of blank and RP samples have only one decomposition peak at 419.1°C and 303.8°C, respectively. That is the decomposition temperature of those reactants, accompanied by a weight loss of 81.3% for blank and 71.2% for RP.

The compatibility of the pyrotechnic composition can be based on comparing the thermal change and mass change of the test samples, as shown in Fig. 9 and Fig. 10. In Fig. 9, it is easy to see that the decomposition peak of sample M0 is larger than the decomposition peak of pure RP. Figure 9 shows that at a temperature of about 300°C, pure RP begins to decompose, and the mass quickly decreases. In the same temperature range, the sample M0 almost does not lose weight until T > 420°C, with a significant weight change. Thus, it can be concluded that the pyrotechnic composition composed of blank and RP is highly compatible according to STANAG 4147 standards.

In addition, sample M0 was also measured in the air to compare with the sample measured in inert gas. The results of the TG/DTA curve of sample M0 in air are shown in Fig. 11.

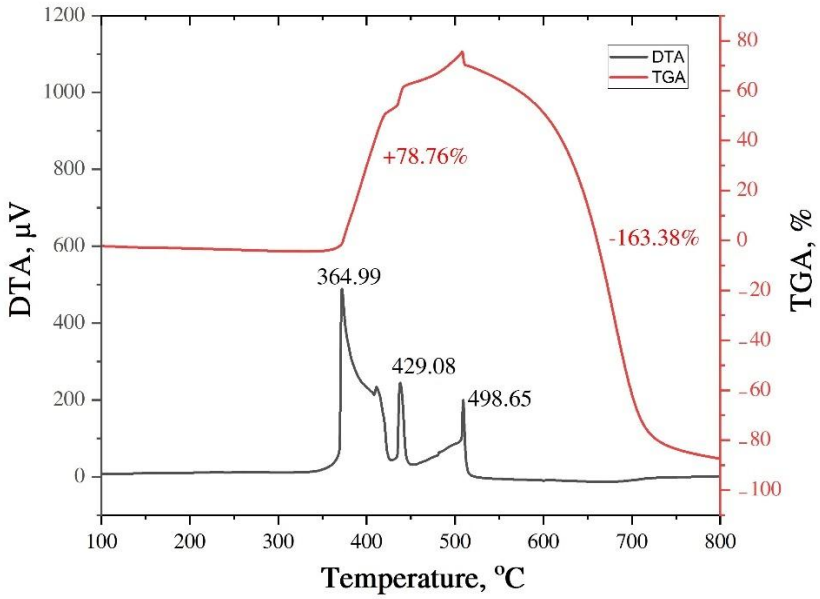

Fig. 11. The TG/DTA curves of M0 under air.

In Fig. 11, the DTA curve of sample M0 has three exothermic peaks at 364.99°C, 429.08°C, and 498.65°C, respectively. Correspondingly, on the TG curve of sample M0, at the peak of 364.99°C, the mass of the sample begins to increase. This can be explained by the fact that $NaNO_3$ starts to decompose and create oxygen to supply the combustion process of RP. The increase in mass and heat release is due to the combustion of RP in oxygen in the air, which are successive phase transitions according to the following reactions [13]:

$$4P_{(red)} + 17.7\,kJ.mol^{-1} \rightarrow P_{4(g)} \quad (1)$$

$$P_{4(g)} + 5O_2 \rightarrow P_4O_{10(s)} + 2988\,kJ.mol^{-1} \quad (2)$$

In addition to the oxygen provided by $NaNO_3$, RP also takes more oxygen from the air, so the mass of the sample increases. This series of reactions forms solid P_4O_{10}. The solid P_4O_{10} can meet moisture and form phosphoric acid H_3PO_4. At the peak of 429.08°C, a secondary reaction may continue to occur, causing the mass to continue to increase. Finally, at the peak of 498.65°C, a combustion reaction and decomposition of other ingredients such as Viton and PTFE occurred.

3.4. Mechanical strength analysis

Samples M1-M4 were compressed into pellets with a height of 20 mm, a diameter of 12 mm, and a density of 1.6 $g.cm^{-3}$. Each test was conducted three times, and the average value was taken. The results are shown in Table 2, Fig. 12, and Fig. 13.

Table 2. The results of mechanical strength tests

Sample	P1, %	Area, mm²	Max Load, kgf	Strength, kgf/mm²	Height, mm	Max Disp., mm	Strain, %
M1	100	113.10	231.10	2.04	18.30	2.62	14.34
M2	90	113.10	256.65	2.27	18.00	2.51	13.96
M3	80	113.10	263.10	2.33	17.90	2.44	13.64
M4	70	113.10	269.75	2.39	14.60	1.71	11.56

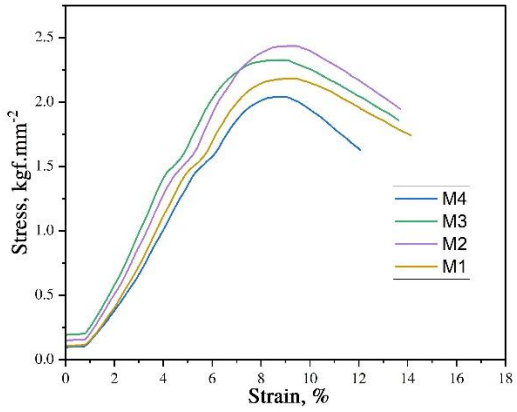

Fig. 12. Stress versus strain curve for M1 ÷ M4.

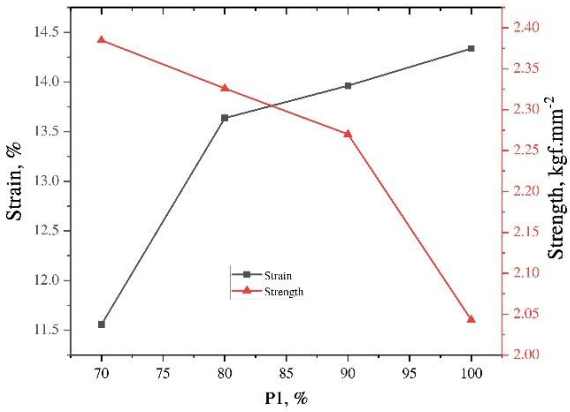

Fig. 13. The strain and strength of M1 ÷ M4.

From Table 2 and Fig. 13, it can be seen that when we increase the amount of free RP outside of the pyrotechnic particles, the compressive strength increases from 2,043 to 2,385 kgf.mm^{-2} and besides that, the strain also decreases from 14,337 to 11,558%. This proves that samples with free RP particles have better compression resistance and lower deformation. This can be explained by free RP particles adhering to the pyrotechnic particles, and when compressed, the pyrotechnic particles have better adhesion. The RP powder may have filled the gaps between the pyrotechnic particles.

The results of measuring the compressive strength indicate that increasing the adhesion and compression resistance of the pellet is achievable by dividing RP into two parts. Part 1 is incorporated before granulation, while part 2 is later mixed with the granules. The content of part 2 (P2) can vary from 10 to 30%.

4. Conclusion

This study studied the technological process of preparing pyrotechnic compositions based on RP and the influence of methods of mixing the RP into pyrotechnic composition on thermal and mechanical characteristics. The results show that the samples have good uniformity with the high compatibility. Sample M1 exhibits the most uniform distribution and reaches a maximum combustion temperature of 1500°C. The mass burning rates of

samples ranging from 0.035 to 0.063 $g.s^{-1}$ and not according to specific rules. Additionally, the compressive strength of samples containing free RP content is higher than that of samples with 100% RP mixed before granulation.

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Tóm tắt: Trong nghiên cứu này, quy trình công nghệ chế tạo thuốc hỏa thuật tạo khói trên cơ sở RP đã được nghiên cứu. Ngoài ra, sự ảnh hưởng của phương pháp trộn RP đến các đặc trưng nhiệt, cơ lý cũng được khảo sát. Các phép đo như: Quang phổ hồng ngoại (FTIR), kính hiển vi điện tử quét (SEM), phân tích thành phần nguyên tố (EDX) được sử dụng để đánh giá mức độ hoàn thiện của các mẫu thuốc; tốc độ cháy, nhiệt độ cháy, phân tích nhiệt TG/DTA được sử dụng để đánh giá các đặc trưng nhiệt; độ bền nén được sử dụng để xác định độ bền nén, ngoài ra, nhiệt độ bùng cháy và độ nhạy ma sát cũng được sử dụng để đánh giá mức độ an toàn của các mẫu thuốc. Kết quả chỉ ra rằng các mẫu thuốc đều có độ hoàn thiện tốt và độ tương thích của các thành phần trong thuốc hỏa thuật là cao. Các mẫu thuốc có RP được trộn sau khi tạo hạt có độ nhạy ma sát và xung nhiệt cao, nhưng lại có độ bền nén tốt hơn mẫu thuốc có 100% RP được trộn trước khi tạo hạt. Các mẫu thuốc có tốc độ cháy khối tương đương nhau nằm trong khoảng 0,035 - 0,063 $g.cm^{-1}$, nhiệt độ cháy của các mẫu thuốc nằm trong khoảng 1100 - 1500^oC.

Từ khóa: *Quy trình công nghệ; hỏa thuật; phốt pho đỏ (RP).*

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