DESIGNING INGREDIENTS OF BTX FLASH-REDUCED PROPELLANT WITH A HIGH PVC CONTENT

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Abstract

Based on experiments, it was researched to determine the kind of pyroxylin and the effect of flash-reduced additives (PVC) on the quality of BTX propellant. Therefore, it designed ingredients for the research and manufacture of propellant of BTX with high PVC content. The results show that the kind of pyroxylin used type BA with a high nitrogen content (about 13.26% N). The flash-reduced additive is the PVC with chlorine content at 61-65% and the thermochemical coefficient of PVC is about -(2 ± 0.5) kcal/(kg × %). When using PVC with the above properties the chemical stability of the propellant is high. At that time, the ingredients of BTX flash-reduced propellant with a high PVC content are determined to be about 77% Pi-BA; 20% PVC; 1.38% DPA; 0.35% ethyl-alcohol; 0.15% ethyl-ether; 1.1% moisture and about 0.02% graphite (addition).

Keywords: *Propellant; BTX; chemical stability; PVC; thermochemical coefficient; chlorine content; ingredients.*

1. Introduction

According to [1]-[3], BTX propellant is a family of flash-reduced propellant. It is a single-base propellant. The symbol BT is the pyroxylin propellant which is used for rifle bullets. The propellant is in grain and cylindrical shape with a hole. Its nominal burning thickness ($2e_1$) is 0.32 mm. The letter "X" is identified as polyvinyl chloride (PVC). The chemical composition of BTX propellant includes: pyroxylin (Pi), PVC, diphenyl-amine (DPA), ethyl-alcohol, ethyl-ether and a very small amount of graphite carbon (C_{gr}). The Pi has role as a main enegy component of propellant, the PVC has role as flash-reduced additive, the DPA has role as chemical stabilizer, ethyl-alcohol and ethyl-ether have role as plasticizers and the C_{gr} has role as anti static-electrical additive. These components present in the propellant about 75-96% Pi, 10-20% PVC, 1-2% DPA, up to about 1% of ethyl-alcohol and ethyl-ether and up to about 0.05% C_{gr}.

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DOI: 10.56651/lqdtu.jst.v3.n01.915.pce

The mechanism of the muzzle flash reduction of PVC compound is determined by the process of propellant burning at high temperature and pressure decomposed PVC to release chlorine gas products in activating form. These gases are combined with substances that were produced by the burning propellant such as CO, H_2, N_2, and CH_4 to create non-flammable products such as $COCl_2$, HCl, CH_2Cl_2 [1], [3]. Beside PVC, potassium sulfate is also used as muzzle flash-reduced additive [4], [5]. However, it is not used for single-base propellant that has a small particle size as a family BT propellant.

In Vietnam, research on flash-reduced propellant of the BTX family has only stopped at the BTX-10 propellant [6]-[8]. The BTX-10 only gives muzzle flash-reduced effective for firings from short to medium barrels. When used for long barrel firings the muzzle flash-reduced effective is decreased or even inefficient.

Ingredients that directly affect the quality of BTX flash-reduced propellant are pyroxylin and PVC. The propellant is used by pyroxylin with suitable nitrogen content. When burning, it will have enough energy to release active chlorine atoms in PVC. Besides, the PVC must also have properties when burning has enough active chlorine atoms but it is not effected on the chemical stability of BTX propellant.

In [6], [9], [10], the authors mentioned BTX-20 propellant. However, in [6], [10] the authors only researched to determine the content of PVC and some properties of thermal decomposition and IR spectrum of PVC. In [9], the authors only researched to determine ingredients of BTX propellant on the basis assumption, because at that time it was not determined the PVC content in BTX-20, simultaneously it considered properties of PVC in BTX-10 similar to PVC in BTX-20. In [7], the authors researched to determine chlorine content in polymer. These documents were not researching to effect of chlorine content in the PVC on chemical stability of propellant. Besides, there were not determined the thermochemical coefficients of PVC which have low chlorine content and high chlorine content. Therefore, researching to effect of chlorine content in the PVC on chemical stability of propellant and choosing suitable pyroxylin on the basis experiment are necessary. Thereby, ingredients of BTX flash-reduced propellant with high PVC content were designed.

2. Research objects and methods

2.1. Research objects

The BTX-20 flash-reduced propellant sample (Russia, taken from 100 mm bullet model 1944). The PVC samples have nominal chlorine content at 55-57%, 61-65%, and 65-69%, respectively. Experimental formulas to calculate heat of combustion, thermochemical coefficient and ingredients of BTX flash reduce propellant with a high PVC content.

2.2. Research methods

Based on the analysis results combined with calculations by experimental formulas or REAL software, it was determined kind of pyroxylin and the effect of PVC on the quality of BTX propellant with high PVC content. The evaluating methods were used to check the features of semi-finished products and products during the research process includes: the method to evaluate the gelling ability of semi-finished products was tested by pressure P_k; the method for determining the shape and size of propellant particles was tested by 06 TCN 836:2000 standard; the method for determining cellulose nitrate content was tested by 31 TC 120:2001 standard; the method for determining diphenylamine content was tested by TCVN/QS 754:2013 standard; the method for determining volatile substances content was tested by TQSA 284:2019 standard; the method for determining the heat of combustion was tested by TCVN/QS 889:2019 standard; the method for determining density was tested by TQSA 1282:2006 standard and the method for determining chemical stability was tested by TQSA 417:2006 standard. The determination of chlorine content in PVC and graphite content in BTX was based on the following principles:

- *Method for determining chlorine content:* Based on the principle of the Etka method (developed by a Russian scientist) for sample decomposition, followed by Mohr titration to determine the chlorine content in the polymer.

Principle of the Etka method: Under the effect of high temperature 500-700°C, the polymer decomposes and releases HCl gas, which then reacts with a mixture of 60% MgO and 40% anhydrous Na_2CO_3 (referred to as the Etka mixture).

$$\text{Polymer} \rightarrow HCl\ (\text{gas}) + CO_2\ (500\text{-}700°C)$$

$$HCl + MgO \rightarrow MgCl_2 + H_2O$$

$$2HCl + Na_2CO_3 \rightarrow 2NaCl + CO_2 + H_2O$$

Sample preparation method: The polymer sample is finely ground into powder with a particle size no larger than 0.45 mm (e.g., PVC and chlorinated PVC powders), then thoroughly mixed with the Etka mixture. The sample is heated in a furnace at a temperature of approximately 500-700°C for 30 to 60 minutes. After cooling, the ashed sample is dissolved in a diluted HNO_3 solution with a concentration of 1.5 N to 3 N (depending on the amount of Etka mixture used). The solution's pH is adjusted to neutral (pH = 7-8) using alkaline and acetic acid solutions, then diluted to a fixed volume in a volumetric flask. The chlorine content in the solution can be determined using various methods; here, the chloride ions (Cl^-) are titrated using the Mohr method.

- *Method for determining the content of PVC and graphite:* Based on the principle that the propellant sample completely dissolves in acid, while PVC and C_{gr} do not dissolve. From this, the total content of PVC and C_{gr} is determined. The solid portion (PVC and C_{gr}) is then dissolved in acetone, in which PVC dissolves and graphite remains insoluble. This allows determination of the C_{gr} content. The remainder is PVC.

2.3. Materials and chemicals

Materials and chemicals are used for making propellant samples: pyroxylin type BA and pyroxylin type CA of Vietnam (VN); diphenylamine - DPA of China (CN); polyvinyl chloride - PVC (VN); ethyl alcohol - C_2H_5OH (CN); ethyl ether - $C_2H_5OC_2H_5$ (CN); polyvinyl chloride with a high chlorine content - PVC (CN) and carbon graphite - C_{gr} (CN). These chemicals and materials meet the requirements for making propellant.

Materials and chemicals are used to analyze chlorine content in PVC and PVC content in BTX: MgO (CN), Na_2CO_3 (CN), NaOH (CN), CH_3COOH (CN), K_2CrO_4 (CN), HNO_3 99% (VN); acetone type AR (CN), pH indicator paper (CN); ceramic cup capacity 300 mL, porous filter funnel capacity 100 mL type G4; vacuum flask; glass cups of all kinds; glass rod; rubber stoppers of all kinds; 250 mL conical flask with ground stopper; distilled water jet tank.

2.4. Equipment and tools

VE125-Vacuum filter pump, binder drying oven, OHOUS electronic scale with model number B229127323, accuracy 0.0001 g, technical electronic scale with accuracy 0.01 g. All types of scales are still under inspection.

Equipment system for making single-base propellant: Centrifugal-type water-removing equipment with a rotating speed of about 300 rpm (capacity is about 4 L), 50-ton hydraulic press equipment, cutting equipment, air reflux ovens, binder drying ovens, graphitization equipment, all kinds of sieves.

Equipment for analysis and measurement: Equipment to measure to test pressure

P_k; high-pressure liquid chromatography (HPLC) equipment to determine DPA content; equipment to measure combustion heat; equipment to determine the solvent content of ethyl alcohol and ethyl ether; equipment to measure the shape and size of propellant; equipment to measure density; equipment to measure IR spectra of PVC; equipment to measure chemical stability of propellant; tools to determine PVC content in BTX propellant and kiln to heat samples and tools to determine chlorine content in PVC.

3. Results and discussion

3.1. Research to determine pyroxylin type in BTX propellant with high PVC content

The BTX flash-reduced propellant is pyroxylin propellant. Depending on the requirements of each different type the raw materials are pyroxylin type BA or pyroxylin type CA. According to [8], [9], the raw material used for the BTX-10 flash-reduced propellant is pyroxylin type CA. Thus, the question of what type of pyroxylin will be used for the BTX flash-reduced propellant with a higher PVC content than BTX-10. This problem needs to be researched and determined.

To determine the type of pyroxylin for the BTX propellant with high PVC content, the author conducted to analyze the components and tested the heat of combustion of the BTX-10 and the BTX-20 samples. The results are shown in Tab. 1.

Tab. 1. Results analyze the BTX-10 and BTX-20 samples

No.	Quantity to be analyzed	Unit	Results	
			BTX-10	BTX-20 [Rus]
1	Content of nitrate cellulose (NC)	%	86.76	77.26
2	Content of diphenylamine (DPA)	%	1.68	1.28
3	Content of external volatile substance	%	1.41	1.31
4	Content of inside volatile substances	%	0.68	0.10
5	Content of PVC	%	9.47	20.01
6	Content of carbon graphite	%	trace	0.04
7	Combustion heat, Q_v	kcal/kg	788.6	730.53

In Tab. 1, the content of PVC and graphite are determined according to [10].

Besides, the infrared spectrum (IR) results of muzzle flash reduce additive in BTX propellant was determined as shown in Fig. 1 [6].

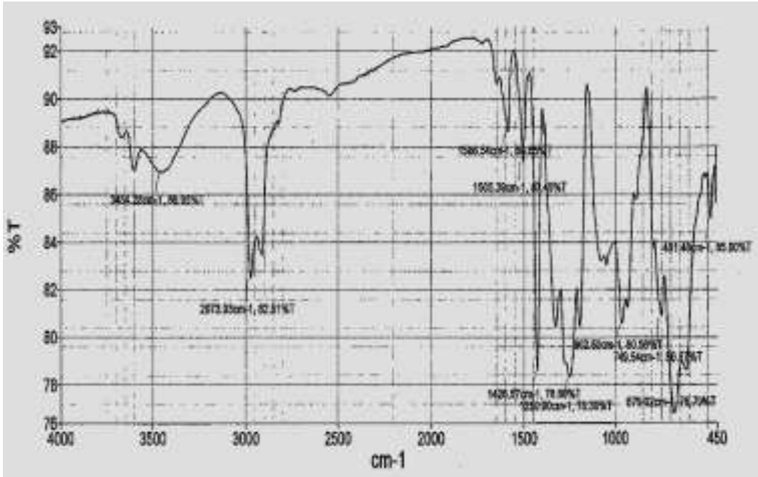
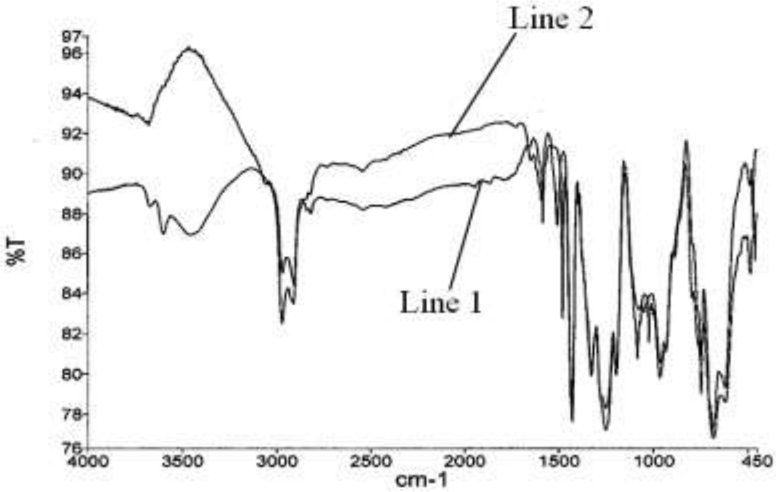


Fig. 1. The infrared spectrum (IR) results of PVC in BTX propellant.

When studying spectral analysis, the author compared the IR spectra of PVC in BTX with the IR spectra of different PVC types. The result shows that the IR spectra of PVC in BTX similar to the IR spectra of PVC with a chlorine content at 61-65%. The results are shown in Fig. 2.



Line 1 - IR spectra of PVC in BTX; Line 2 - IR spectra of PVC with a chlorine content at 61-65%

Fig. 2. Comparison of IR spectra of PVC in BTX and PVC with a chlorine content at 61-65%.

Figure 2 shows that the special peaks of PVC in BTX are completely similar to the special peaks of PVC with a chlorine content at 61-65%. The characteristic peaks are observed at 749, 963, 1251, 1427, 1586, and 2974 cm^{-1}.

The kind of pyroxylin materials of BTX-20 flash-reduced propellant is determined based on the heat of combustion and experimental formulas.

According to [1], [3], calculation of the combustion heat and the thermochemical coefficient and different parameters of flash-reduced propellant are determined based on the experimental formulas (1), (2), (3), and (4) as follows:

$$Q_v = \sum m_i \cdot \beta_i \tag{1}$$

where m_i is content of ingredient i in that compound, %; β_i is thermochemical coefficient of ingredient i, kcal/kg × %.

The thermochemical coefficient is determined experimentally or it can be calculated by the formula:

$$\beta = \frac{(285 \cdot n_{(o)} - 174 \cdot n_{(C)} + \Delta H)}{100M}, \text{ kJ/kg} \times \% \tag{2}$$

where $n_{(O)}$, $n_{(C)}$ are the number of oxygen and carbon atoms in 1 mole of substance, respectively; ΔH is heat of formation of the substance, kJ/kg; M is molar mass, kg/mol.

With NC, the thermochemical coefficient is calculated according to the Asobu formula as follows:

$$\beta = 1.3 \cdot N - 6.7, \text{ kJ/kg} \times \% \tag{3}$$

where N is the nitrogen content in pyroxylin. It is calculated according to the conversion formula from the gas equation of state as follows:

$$N = \frac{14 \cdot V_{\text{mL NO/g}}}{22.4 \cdot 1000} \cdot 100, \% \tag{4}$$

where $V_{\text{mL NO/g}}$ is the nitrogen oxide content in pyroxylin, mL NO/g.

The thermochemical coefficients of the substances are given in Tab. 2.

Table 1 and 2 combined with formulas (1), (2), (3), and (4) can be calculated the thermochemical coefficient of PVC, β = -2.17 kcal/(kg × %).

On the other hand, if symbol the thermochemical coefficient of pyroxylin material is β_{Pi}, it will have formula (1) as follows:

$$Q_v = \sum m_i \times \beta_i \tag{5}$$

Transfer parameters selected from Tab. 1 to formula (5) gives:

$$730.53 = 77.26(\beta_{Pi}) + 1.28(-29) + 0.04(-17.5) + 0.06(-22.5) + 0.04(-30) + 20.01(-2.17).$$

Solving the above mathematical equation:

β_{Pi} = 10.54 kcal/(kg × %)

This value is replaced by formula (3) when the nitrogen content in the pyroxylin is 13.26% corresponding to pyroxylin type BA.

Tab. 2. Thermochemical coefficients of substances

No.	Substances	β, kcal/(kg × %)	Note
1	NC	12.88	Content of N in NC is 204.79 mL NO/g
2	DPA	-29.0	-
4	PVC	-	Unknown, it is needed to determine
5	Graphite	-30.0	-
6	H_2O	0.0	-
7	C_2H_5OH	-17.5	-
8	$(C_2H_5)_2O$	-22.5	-

It can be seen that the use of up to 20% PVC in BTX flash-reduced propellant must use pyroxylin type BA with content of 13.26% N (required from 13.09% to 13.25% N). Thus, if it still uses pyroxylin type BA the above giving the BTX propellant sample but increases the PVC content up to 30% (such as the BTX-30), when the heat of combustion will decrease about 130 kcal/kg corresponding to the heat of combustion value of about 600 kcal/kg for the BTX-30 flash-reduced propellant. This heat of combustion is difficult to ensure complete combustion of BTX-30 propellant. On the other hand, when using 30% PVC, it will not ensure the ability to gel the semi-finished propellant after the compression stage.

The above results can be seen that the highest PVC content is about 20%.

3.2. Research in determine the thermochemical coefficient of PVC

Currently, there are three commercially popular product types of PVC such as regular PVC with a chlorine content is at 55-57%, the chlorinated PVC type with a chlorine content is at 61-65%, and the chlorinated PVC type with a chlorine content is at 65-69%. To determine their application characteristics in propellant, it is first necessary to determine the thermochemical coefficient on the basis to make BTX flash-reduced propellant samples. Where sample of M1.BTX contains PVC with a chlorine content at 55-57%, sample of M2.BTX contains PVC with a chlorine content at 61-65%, and sample of M3.BTX contains PVC with a chlorine content at 65-69%. Then, it tested substances of propellant samples. The results are shown in Tab. 3.

Tab. 3. Results of analysis BTX flash reduce propellant samples

No.	**Quantity to be analyzed**	**Unit**	**Results**		
			M1.BTX	**M2.BTX**	**M3.BTX**
1	Content of NC	%	77.06	77.44	77.04
2	Content of DPA	%	1.31	1.27	1.24
3	Content of external volatile substance	%	1.18	1.01	1.14
4	Content of inside volatile substances	%	0.31	0.30	0.51
5	Content of PVC	%	20.14	19.98	20.07
6	Content of carbon graphite	%	trace	trace	trace
7	Heat of combustion, Q_v	kcal/kg	604.75	731.71	765.01

Table 1 data and Tab. 2 data (where pyroxylin type BA is used at 13.2% N) combined with formulas (1), (2), (3), and (4) can be calculated the thermochemical coefficient of PVC samples in M1.BTX, M2.BTX, and M3.BTX respectively as follows: $\beta_{PVC.M1}$ = -8.01 kcal/(kg × %), $\beta_{PVC.M2}$ = -1.94 kcal/(kg × %), and $\beta_{PVC.M3}$ = 0.08 kcal/(kg × %).

The results of Tab. 3 combined with the above calculations show that PVC in BTX-20 flash-reduced propellant has a thermochemical coefficient of about -1.94 kcal/(kg × %).

Compared to the calculating results in Section 3.1, the thermochemical coefficient of PVC in BTX flash-reduced propellant has a small difference (Section 3.1, β = -2.17 kcal/(kg × %). The author's calculations are based on experimental data. On the other hand, even PVC itself when chlorinated to make product is not uniform in its molecular structure. Therefore, the difference as mentioned is completely acceptable. Thus, the thermochemical coefficient of PVC in BTX flash-reduced propellant is determined β = -(2 ± 0.5) kcal/(kg × %).

3.3. Effect of chlorine content in PVC on the chemical stability of BTX propellant

Based on [7], the author determined the reality chlorine content in the PVC samples which is used to make the BTX samples in Section 3.2. The results are shown in Tab. 4.

Tab. 4. Result of chlorine content in PVC

No.	**Quantity to be analyzed**	**Unit**	**Results**		
			M1.PVC	**M2.PVC**	**M3.PVC**
1	Reality chlorine content in PVC sample	%	56.16	62.23	66.34

Table 4 shows that the reality chlorine content in the PVC samples is 56.16% for the regular PVC sample with a chlorine content at 55-57% (M1.PVC), 62.23% for chlorinated PVC sample with a chlorine content at 61-65% (M2.PVC), and 66.34% for chlorinated PVC sample with a chlorine content at 65-69% (M3.PVC).

To study the effect of chlorine content on the chemical stability of BTX propellant, the author conducted measurements chemical stability of propellant samples by the Vieille method. The results are shown in Tab. 5.

Tab. 5. Relationship between chlorine content in PVC and chemical stability of BTX

No.	**Quantity to be analyzed**	**Unit**	**Results**		
			M1.BTX	**M2.BTX**	**M3.BTX**
1	Chemical stability by the Vieille method	hour	66	65	58

Note: M1.BTX sample is used by PVC with 56.16% chlorine, M2.BTX sample is used by PVC with 62.23% chlorine and M3.BTX is used by PVC with 66.34% chlorine.

Table 4 and Tab. 5 show when it increases the chlorine content in PVC from 56.16% to 62.23% the chemical stability of BTX propellant does not decrease much (65 hours compared to 66 hours). However, if the chlorine content increases to 66.34% its chemical stability decreases significantly (58 hours compared to 66 hours).

The increase/decrease at chlorine content in PVC leading to a change in chemical stability can be explained that the Cl atom at the H position (replaced by Cl) in the PVC molecule will be less stable than the Cl atom in the PVC monomer as well as in the PVC molecule. Therefore, when the PVC is decomposed by temperature, the activating Cl atoms in PVC molecules which have a high chlorine content will be released faster than comparing to PVC which has a low chlorine content. When chlorine content is not saturated as PVC with a chlorine content at 61-65%, may not be affect the chemical stability of the propellant. When the chlorine content in PVC molecules reaches limitation the PVC will effect on the chemical stability of the propellant.

Research results in Section 3.2 and Section 3.3 show that the PVC used in BTX flash-reduced propellant has a chlorine content in the range from 61% to 65%.

3.4. Ingredients design of BTX flash-reduced propellant with a high PVC content

To design ingredients of BTX flash-reduced propellant with a high PVC content needs to choose the kind of pyroxylin type BA (with a high nitrogen content, about 13.20% N) and the PVC has a chlorine content at 61-65% with its thermochemical coefficient, $\beta = -(2 \pm 0.5)$ kcal/(kg × %). There are also other ingredients such as diphenyl-amine, carbon graphite and some plasticizing solvents such as ethyl-alcohol and ethyl-ether. These ingredients must ensure a ratio so that the heat of combustion is from 725 kcal/kg to 735 kcal/kg.

Based on the above request and input data combined with REAL software, it can be calculated to determine the content range of ingredients to make a propellant sample that the heat of combustion achieves as required. The results are shown in Tab. 6.

In fact, when using the REAL software, the PVC has not in the REAL software data so the PVC is replaced by DNT. The thermochemical coefficient of DNT is about zero kcal/(kg × %) while the thermochemical coefficient of PVC is about -2.0 kcal/(kg × %). Therefore, the reality heat of combustion value will have to be subtracted about 40 kcal/kg compared to the results in Tab. 6 and when the combustion heat of samples will be the value of 710; 722; 731; and 738 kcal/kg, respectively. Where only the value of 731 kcal/kg meet the requirements from 725 kcal/kg to 735 kcal/kg. Therefore, the ingredients of the BTX flash-reduced propellant sample with a high PVC content should be designed as S3.BTX sample shown in Tab. 6.

Tab. 6. Calculating results of kinematics parameters of propellant samples

No.	Ingredients and kinematics parameters	Value			
		S1.BTX	S2.BTX	S3.BTX	S4.BTX
Ingredients, %					
1	NC with nitrogen content as 13.20% N	75.0	76.0	77.0	78.0
2	DPA	1.38	1.38	1.38	1.38
3	PVC	22.0	21.0	20.0	19.0
4	Graphite	0.02	0.02	0.02	0.02
5	Ethyl alcohol	0.35	0.35	0.35	0.35
6	Ethyl ether	0.15	0.15	0.15	0.15
7	External volatiles (H_2O)	1.10	1.10	1.10	1.10
Kinematics parameters (at here the DNT is replaced by PVC)					
1	Force of propellant, kJ/kg	940482	948954	960518	965883
2	Temparature of combustion, K	2452	2514	2564	2611
3	Heat of combustion, kcal/kg	750	762	771	778
4	Gas volume, L/kg	1028	1015	1006	1001
5	Burning rate coefficient	1.11×10^{-9}	1.15×10^{-9}	1.18×10^{-9}	1.21×10^{-9}

4. Conclusion

It researched to determine the kind of pyroxylin and the effect of chlorine content in PVC on the chemical stability of BTX flash-reduced propellant. The results show that the kind of pyroxylin is used by type BA with a high nitrogen content (about 13.26% N). The PVC with a chlorine content at 61-65% has the chemical stability similar to regular PVC with a chlorine content at 55-57% and to give the chemical stability of BTX propellant better than comparing to the PVC with a chlorine content higher than of 65%. The PVC that used for BTX flash-reduced propellant has the thermochemical coefficient of about -(2 ± 0.5) kcal/(kg × %) and when the ingredients of BTX flash-reduced propellant with a high PVC content are determined as follows: 77% Pi-BA, 20% PVC, 1.38% DPA, 0.35% ethyl-alcohol, 0.15% ethyl-ether, 1.1% H_2O, and 0.02% graphite (addition).

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THIẾT KẾ ĐƠN THÀNH PHẦN CỦA THUỐC DẬP LỬA HỌ BTX CÓ HÀM LƯỢNG PVC CAO

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Tóm tắt: Trên cơ sở thực nghiệm, đã tiến hành nghiên cứu xác định được loại nguyên liệu pirốcxilin và ảnh hưởng của phụ gia dập lửa (PVC) đến chất lượng của thuốc phóng BTX. Qua đó, đã xác lập được đơn thành phần phục vụ cho việc nghiên cứu, chế tạo thuốc dập lửa BTX có hàm lượng PVC cao. Kết quả cho thấy, loại nguyên liệu pirốcxilin đã sử dụng là mác BA có hàm lượng nitơ cao (khoảng 13,26% N), phụ gia dập lửa là PVC với hàm lượng clo từ 61% đến 65% và hệ số nhiệt lượng cháy của PVC khoảng -(2 ± 0,5) kcal/(kg × %). Khi sử dụng PVC với đặc tính đã nêu cho độ an định hóa học Vi-ây của thuốc phóng cao. Khi đó, đơn thành phần được xác lập cho mác thuốc dập lửa BTX có hàm lượng PVC cao là khoảng 77% Pi-BA; 20% PVC; 1,38% DPA; 0,35% cồn êtylic; 0,15% ête êtylic; 1,1% ẩm và ngoài ra có khoảng 0,02% graphit.

Từ khóa: *Thuốc phóng; BTX; an định hóa học; PVC; nhiệt lượng; hàm lượng clo; đơn thành phần.*

Received: 02/12/2024; Revised: 16/04/2025; Accepted for publication: 28/04/2025

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