

Research on the phlegmatization technology of RDX with oxidized polyethylene wax

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ABSTRACT

In this paper, several factors affecting the preparation of A-IX-10 explosive by a surface modification method in an aqueous emulsion system using oxidized polyethylene wax (OPE wax) dissolved in solvent cyclohexane and octylphenol ethoxylate (Triton X-100) surfactant were investigated. The results showed that replacing oxyzin wax with OPE wax at a dissolution concentration in cyclohexane solvent not exceeding 5% and employing Triton X-100 surfactant at low concentrations not exceeding 0.1 wt.% relative to hexogen (RDX), improves the coating coverage of the OPE wax layer on the surface of RDX particles. As a result, the obtained particles were fine, free-flowing, uniform in size, and free from agglomeration. The A-IX-10 explosive prepared in this study exhibited technical characteristics comparable to those of Russian A-IX-10 explosive, but with significantly lower impact sensitivity.

Keywords: Explosive phlegmatization; A-IX-10 explosive; Oxidized polyethylene wax; Triton X-100 surfactant.

1. INTRODUCTION

A-IX-10 (Gekfol-5) is a highly phlegmatized explosive with a composition similar to that of A-IX-1, consisting of 93.5–95 wt.% RDX and 5–6.5 wt.% phlegmatizer, the latter comprising 98% oxyzin and 2% organic pigment [1]. It is used in several Russian munitions, including S8-KO and S8-KOM aircraft rocket warheads and 9N210 submunitions in the 9M27K uragan multiple launch rocket system (MLRS) cluster warhead.

Oxyzin is essentially an oxidized ceresin wax developed to improve adhesion between explosive particles and metallic surfaces compared with unmodified ceresin. A U.S. patent [2] describes the oxidation of ceresin wax to produce a harder oxidized wax. Consequently, compared with A-IX-1, whose phlegmatizer consists of 60% natural ceresin, 38.8% stearin, and 1.2% Sudan dye [1, 3], A-IX-10 is expected to exhibit improved adhesion and hardness. These properties make it more suitable for warheads subjected to high launch accelerations, reducing the risk of charge cracking and helping to maintain warhead performance.

In addition, the natural ceresin used in A-IX-1 has a melting point of 77–78 °C [4], which is lower than that of oxyzin used in A-IX-10 (88–97 °C) [5]. The use of a higher-melting-point phlegmatizer extends the operational temperature range of the warhead. However, securing a reliable supply of oxyzin and developing domestic production capability remain challenging.

Oxidized polyethylene wax (OPE wax) possesses physicochemical properties similar to those of oxyzin, including a comparable melting point (~103 °C) and the presence of polar functional groups such as carboxyl, carbonyl, and hydroxyl groups. Owing to these characteristics, OPE wax is widely used as a coating and dispersing agent in polymer processing [6].

Therefore, investigating OPE wax as a substitute for oxyzin wax in A-IX-10 explosive is of considerable interest and, to the authors' knowledge, has not yet been reported in Vietnam.

2. EXPERIMENTAL

2.1. Material

Military-grade RDX explosive was milled to a median particle size (D50) of 53.98 µm,

oxidized polyethylene wax (OPE wax, Russia), triethanolamine (USA), stearic acid (India), polyethylene glycol 400 (PEG 400, Russia), Triton X-100 (USA), dioctyl adipate (Russia), and Sudan 3 dye (India).

2.2. Specimen preparation

The phlegmatizer components (OPE wax, dioctyl adipate, and stearic acid) together with Sudan 3 dye were completely dissolved in cyclohexane at 45–50 °C to obtain solutions with concentrations ranging from 0.625 to 10 wt.%. In a separate reactor, RDX was dispersed in water and mechanically stirred at 45–50 °C in the presence of surfactants, including triethanolamine/stearic acid (1:2), PEG 400, and Triton X-100, at concentrations of 0.01–5 wt.% relative to the RDX mass. The phlegmatizer solution was then slowly added to the RDX suspension. After homogenization, the mixture was heated to 60–95 °C and maintained for 30–110 min under stirring at 100–500 rpm. The resulting suspension was cooled, vacuum-filtered and dried.

2.3. Analytical methods

The technical specifications of the A-IX-10 explosive were evaluated in accordance with procedure TPTN.A-IX-10.QTPT.01, with the main equipment used as follows: MS-204 analytical balance; B180 muffle furnace; Vacuum drying oven; EZ-melt melting point apparatus; CAST impact sensitivity tester; Soft-wire ballistic pendulum apparatus for work capacity determination; Lead block compression test apparatus; Kontinitro SA EXPLOMET 2 detonation velocity measurement system; JMS-6510LV scanning electron microscope with energy-dispersive X-ray spectroscopy (SEM-EDX); STABIL vacuum stability tester.

3. RESULTS AND DISCUSSION

3.1. Effect of surfactants on the dispersion of RDX

Since RDX is a hydrophobic explosive, it readily agglomerates or undergoes phase separation when stirred in water. To reduce the interfacial tension between water and the RDX particle surface and thereby improve dispersion, the use of surfactants is essential. In addition to enhancing dispersion, surfactants act as emulsifying agents for the oil phase (phlegmatizer solution) in the aqueous phase, serving as an intermediate bridge between the wax and the RDX particle surface while ensuring high chemical stability and thermal resistance under phlegmatization conditions.

Three surfactant systems commonly used for RDX explosives were selected: triethanolamine/stearic acid (1:2), polyethylene glycol (PEG 400), and octylphenol ethoxylate (Triton X-100). The surfactant-to-RDX mass ratios investigated were 0.01%, 0.05%, 0.1%, 1%, 2%, 3%, and 5%. To ensure safe operation during the phlegmatization process and maintain complete immersion of the impeller in the suspension, the water volume was fixed at 12 L. The dispersion results are presented in Table 1.

Table 1. Effect of surfactants on RDX dispersion.

No.	Surfactant system	Mass ratio relative to RDX, %							
		0,01	0,05	0,1	0,5	1	2	3	5
1	Triethanolamine/stearic acid (1/2)	-	-	-	-	-	+	+	-
2	PEG 400	-	-	-	-	+	+	-	-
3	Triton X-100	-	+	+	-	-	-		

Note: “–” indicates unstable dispersion; “+” indicates stable dispersion.

From the results presented in Table 1, each surfactant system exhibits different dispersion efficiencies. To obtain a stable RDX suspension in water, the minimum surfactant-to-RDX mass ratios required were 2% for the triethanolamine/stearic acid (1:2) system, 1% for PEG 400, and only 0.05% for Triton X-100 (corresponding to 0.0195 wt% in aqueous solution), indicating its

superior dispersing performance. This is attributed to the fact that Triton X-100 surfactant molecules can readily self-assemble into micelles at relatively low concentrations, with a reported critical micelle concentration (CMC) of approximately 0.2–0.3 mM (corresponding to ~ 0.013–0.02 wt% in aqueous solution) [7]. This value is consistent with the experimental concentration employed in this study.

However, Triton X-100 at concentrations above 0.1% produced excessive foaming and significantly increased the suspension viscosity, which hindered visual observation and could limit the dispersion of the phlegmatizer solution. Similarly, concentrations above 3% for the triethanolamine/stearic acid (1:2) system and above 2% for PEG 400 resulted in highly viscous suspensions, reducing the effectiveness of the phlegmatizer coating process. Based on these results, Triton X-100 concentration of 0.05 wt% was selected for subsequent investigations due to its high efficiency and lower cost compared with the other two surfactant systems.

3.2. Effect of phlegmatizer composition on the properties of A-IX-10

According to published data, the phlegmatizer content in Russian A-IX-10 explosive ranges from 5.0 to 6.5 wt.% [1]. Therefore, total initial phlegmatizer contents of 5%, 6%, and 7% were selected for investigation. It is known that the presence of dioctyl adipate (DOA) and stearic acid improves the coverage of the phlegmatizer on the RDX particle surface, reduces sensitivity, and enhances compressibility [5]. Therefore, in addition to OPE wax, DOA and stearic acid were also selected for evaluation. The formulations investigated and the corresponding results are presented in Tables 2 and 3.

Table 2. Investigated formulations with varying phlegmatizer compositions.

No.	Component	Form 1	Form 2	Form 3	Form 4	Form 5	Form 6	Form 7	Form 8
1	RDX	93	94	94	94	94	94	94	95
2	Triton X-100	0,05	0,05	0,05	0,05	0,05	0,05	0,05	0,05
3	OPE wax	6,8	5,8	3,8	3,8	3,8	3,8	3,8	4,8
4	DOA	-	-	2	1,5	1	0,5	-	-
5	Stearic acid	-	-	-	0,5	1	1,5	2	-
6	Sudan 3	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,15

Table 3. Effect of phlegmatizer composition on the physical properties of A-IX-10.

No.	Sample	Analyzed phlegmatizer content (%)	State of A-IX-10 after thermal conditioning at 80 °C for 1 hour
1	Form. 1	6.71	Slightly agglomerated; high inter-particle adhesion
2	Form. 2	5.89	Slightly agglomerated; high inter-particle adhesion
3	Form. 3	5.82	Strongly agglomerated; extreme inter-particle adhesion
4	Form. 4	5.80	Strongly agglomerated; extreme inter-particle adhesion
5	Form. 5	5.79	Strongly agglomerated; extreme inter-particle adhesion
6	Form. 6	5.77	Strongly agglomerated; extreme inter-particle adhesion
7	Form. 7	5.75	Strongly agglomerated; extreme inter-particle adhesion
8	Form. 8	4.83	Slightly agglomerated; high inter-particle adhesion

The results presented in Table 3 show that the addition of DOA and stearic acid (formulations 3–7) softened the A-IX-10 explosive but also caused excessive stickiness, which could significantly affect its mechanical properties during service. In contrast, formulations containing only OPE wax (formulations 1, 2, and 8) exhibited physical characteristics similar to those of the Russian A-IX-10 explosive, which likewise employs only oxyzin wax as the phlegmatizer. However, the phlegmatizer contents of Formulations 1 and 8 were outside the recommended range of 5.0–6.5 wt.%. Formulation 2 yielded a phlegmatizer content of 5.89 wt.%, which falls within this range and closely matches the measured value of 5.93 wt.% for the Russian A-IX-10 sample.

Therefore, an initial phlegmatizer content of 6 wt.% was considered appropriate, and formulation 2 was selected as the optimum formulation for subsequent investigations.

3.3. Effect of solvent-to-water ratio on the properties of A-IX-1O

To select an appropriate amount of cyclohexane for the phlegmatization process of A-IX-1O explosive, the influence of different solvent-to-water ratios (1/4, 1/2, 1/1, 2/1, and 3/1) was investigated, corresponding to phlegmatizer dissolution concentrations in the solvent of 10%, 5%, 2.5%, 1.25%, and 0.625%, respectively. The effects of these ratios on solvent removal time and the physical state of the explosive after vacuum filtration were evaluated. The solvent-removal temperature was selected based on the boiling point of cyclohexane (80 °C). The results are presented in Table 4.

Table 4. Effect of solvent-to-water ratio on the properties of A-IX-1O.

No.	Solvent/water volumetric ratio	Evaporation time (min)	Post-filtration morphological state
1	1:4	50	Slightly agglomerated, coarse particles
2	1:2	70	Highly porous, uniformly fine particles
3	1:1	110	Highly porous, uniformly fine particles
4	2:1	130	Highly porous, uniformly fine particles
5	3:1	150	Highly porous, uniformly fine particles

The results in Table 4 show that the solvent-to-water ratio significantly affects the solvent removal time. As the solvent-to-water ratio increases, the solvent removal time becomes longer. At a ratio of 1/4, the A-IX-1O explosive after vacuum filtration exhibited agglomeration and a larger particle size compared to the 1/2 ratio, where the product was fine, uniform, and well-dispersed. This indicates that at the 1/4 ratio, the lower solvent content and faster evaporation negatively affect the coating efficiency of the phlegmatizer, leading to localized agglomeration. At ratios higher than 1/2, no significant changes in the state of post-filtration of the product were observed. Therefore, a solvent-to-water ratio of 1/2 was selected as optimal due to its balance between product quality and solvent consumption.

3.4. Effects of temperature, evaporation time, and agitation speed on A-IX-1O properties

After optimizing the formulation and the solvent-to-water ratio, the effects of temperature, reaction time, and stirring speed on the properties of A-IX-1O explosive were further investigated. The results of these studies are presented in Tables 5, 6, and 7.

Table 5. Effect of solvent evaporation temperature on the properties of A-IX-1O.

No.	Temperature (°C)	Evaporation time (min)	Post-filtration morphological state
1	60	180	Highly porous, uniformly fine particles
2	70	120	Highly porous, uniformly fine particles
3	80	70	Highly porous, uniformly fine particles
4	90	35	Slightly agglomerated, non-uniform particles
5	95	20 (boil-over observed)	Heavily agglomerated, coarse particles

The composite data from tables 5, 6, and 7 establish that the optimal parameters for the solvent evaporation phase are a temperature of 80 °C, an evaporation time of 70 minutes, and an agitation speed of 300 rpm. Lower temperatures drastically prolong the evaporation process, whereas elevated temperatures induce premature solvent escape, causing rapid particle coagulation. Notably, at 95 °C, a boil-over phenomenon occurs due to the aqueous phase nearing its boiling point. Insufficient evaporation times leave residual solvent within the matrix, causing severe plasticization and clumping. Conversely, excessive processing times offer no morphological improvements but promote significant adhesion of the energetic material to the reactor walls, complicating product recovery and subsequent equipment decontamination. Regarding agitation,

inadequate speeds result in poor emulsification, leading to mild phase separation or color inhomogeneity. Excessive speeds induce severe splashing, which negatively impacts the final formulation stoichiometry and complicates reactor maintenance between batches.

Table 6. Effect of solvent evaporation time on the properties of A-IX-10.

No.	Time (min)	Suspension state	Post-filtration morphological state
1	30	Slightly viscous/gel-like	High residual solvent; heavily plasticized/sticky, difficult to filter
2	50	Slightly agglomerated	Low residual solvent; slightly sticky, coarse particles
3	70	Uniformly dispersed	Trace residual solvent; highly porous, uniformly fine particles
4	90	Slight wall adhesion	Trace residual solvent; highly porous, uniformly fine particles
5	110	Significant wall adhesion	Trace residual solvent; highly porous, uniformly fine particle

Table 7. Effect of agitation speed on the properties of A-IX-10.

No.	Agitation speed (rpm)	Suspension state	Post-filtration morphological state
1	100	Slight phase separation	Agglomerated, coarse particles
2	200	Color inhomogeneity	Slightly agglomerated, uneven coloration
3	300	Uniformly dispersed	Highly porous, uniformly fine particles
4	400	Slight splashing onto reactor walls	Highly porous, uniformly fine particles
5	500	Severe splashing onto reactor walls	Highly porous, uniformly fine particles

3.5. Physicochemical and explosive performance characterization of A-IX-10

Upon finalizing the optimized process parameters, samples of the synthesized A-IX-10 were rigorously characterized for their physicochemical attributes and ballistic performance. These empirical data were subsequently benchmarked against the standard Russian A-IX-10 explosive. The comparative analytical results are provided in Table 8.

Table 8. Technical specifications of the synthesized A-IX-10 explosive.

No.	Parameter	Unit	A-IX-10 (Russia)	A-IX-10 (Vietnam)
1	Appearance	—	Uniform orange granules; no visible impurities	Uniform orange granules; no visible impurities
2	Phlegmatizer content	%	5.93	5.89
3	RDX content	%	94.06	94.08
4	Melting point	°C	203.9	203.8
5	Moisture and volatile matter	%	0.01	0.03
6	Acidity (as H ₂ SO ₄)	%	0.00	0.00
7	Ash content	%	0.10	0.05
8	Acetone-insoluble residue	%	0.58	0.33
9	Impact sensitivity (Cast fall-hammer test)	%	44	12
10	Working capacity (ballistic pendulum test)	% TNT	127.8	128.5
11	Detonation velocity at a density of 1.6 g/cm ³ (extrapolated)	m/s	8157	8153
12	Brisance (Lead cylinder compression test, 25 ± 0.01 g)	mm	18.69	18.45
13	Thermal stability at 100 °C for 40 hours	cm ³ /g	0.080	0.118

Analysis of the results presented in Table 8 shows that, overall, the A-IX-1O explosive prepared by the research group exhibits technical characteristics comparable to those of the Russian A-IX-1O explosive, while displaying significantly lower impact sensitivity. This suggests that the excellent coating ability of OPE wax on the RDX particle surface contributes to improved sensitivity performance, as will be further demonstrated in section 3.6. After applying the optimized processing parameters, the resulting A-IX-1O explosive exhibited fine, free-flowing particles with a high degree of uniformity, as shown in Figure 1.



Figure 1. Morphological appearance of the synthesized A-IX-1O explosive:
(a) post-filtration; (b) post-drying.

3.6. Evaluation of the OPE wax coating efficiency on RDX particles

The A-IX-1O explosive prepared under the optimized phlegmatization parameters was subjected to surface morphological and elemental analyses to evaluate the coating efficiency of the OPE wax layer on the RDX crystals. The surface morphology and microstructural features of the coated particles were examined using Scanning Electron Microscopy (SEM), as depicted in Figure 2. Concurrently, the elemental composition and spatial distribution across the particle surfaces were mapped utilizing Energy-Dispersive X-ray Spectroscopy (EDX), as illustrated in Figure 3.

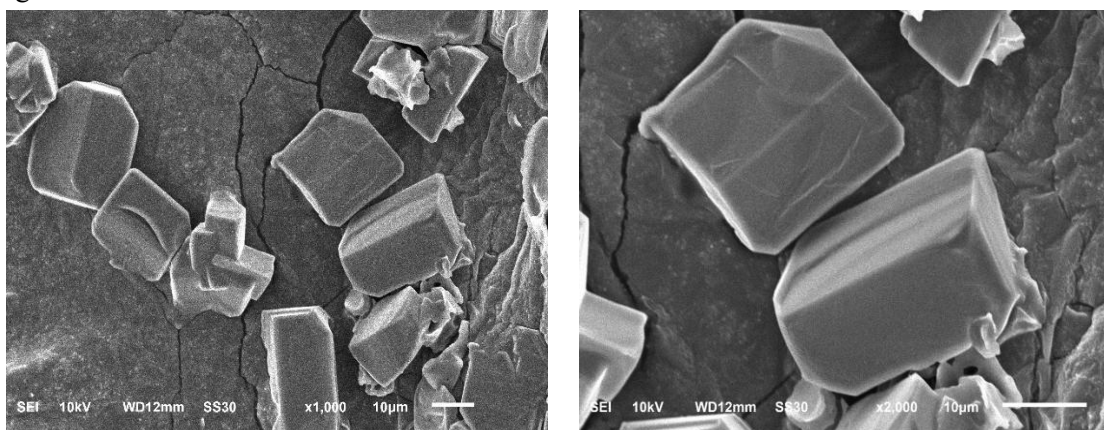


Figure 2. SEM micrograph of the synthesized A-IX-1O explosive surface.

Based on SEM images, the surface of RDX appears to be coated with a relatively uniform and stable wax layer. According to the EDX analysis results, nitrogen was no longer detected in the surface distribution regions of the RDX particles. This result serves as indirect evidence and is consistent with the hypothesis that the OPE wax layer has been deposited on the surface of the

RDX particles. However, these methods are not sufficient to directly confirm the continuity or the degree of surface coverage of the OPE wax coating.

Since the RDX particles used were ground to an average particle size of $53.98\ \mu\text{m}$, which is equivalent to the RDX particle size after removal of the Russian phlegmatizing agent ($53.50\ \mu\text{m}$). However, the RDX particles ground by the research group exhibited relatively pronounced angular features, as shown in the SEM image (Figure 2). It is known that the angular shape of explosive particles is closely related to the formation of hot spots and has higher mechanical sensitivity compared to particles with a rounded shape [8]. Therefore, the inert wax layer uniformly coating the surface of the explosive particles contributes to slowing down or hindering reactions initiated by hot spots formed through the interaction between impact loading and the microstructure of the material [9]. In addition, the wax layer also acts as a lubricant, significantly reducing the friction coefficient and the friction-induced temperature rise between explosive particles [10, 11]. Owing to these factors, the A-IX-10 explosive prepared by the research group exhibits relatively low explosive sensitivity.

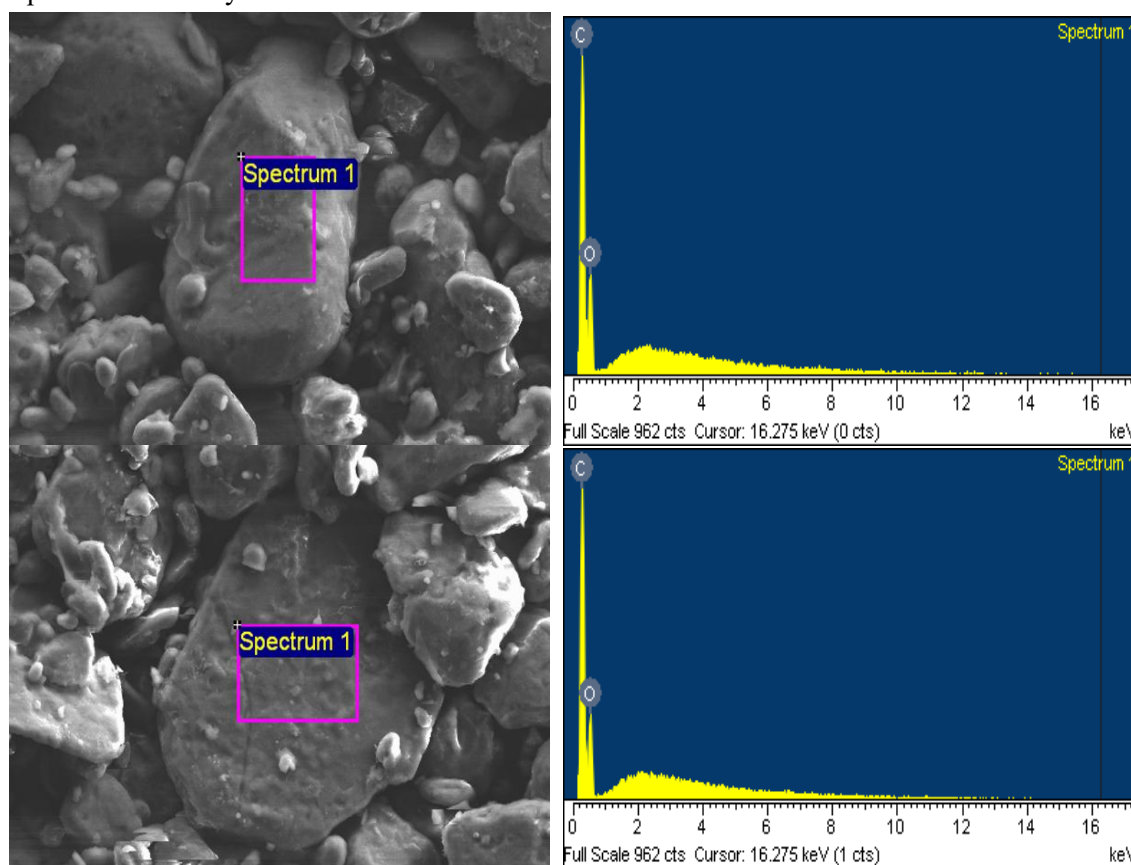


Figure 3. EDX elemental spectrum of the synthesized A-IX-10 explosive surface.

4. CONCLUSIONS

Based on the results obtained, replacing oxyzin with OPE wax at a dissolution concentration in cyclohexane solvent not exceeding 5% and employing Triton X-100 surfactant at low concentrations not exceeding 0.1 wt.% relative to hexogen (RDX), improves the coating coverage of the OPE wax layer on the surface of RDX particles, as confirmed by SEM observations and EDX surface elemental analysis. Furthermore, optimization of the solvent-removal conditions ($80\ ^\circ\text{C}$, 70 min, and 300 rpm) produced A-IX-10 particles that were fine, free-flowing, uniform in

size, and free from agglomeration. The A-IX-1O explosive prepared in this study exhibited technical characteristics comparable to those of Russian A-IX-1O explosive, but with significantly lower impact sensitivity.

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TÓM TẮT

Nghiên cứu công nghệ thuần hóa thuốc nổ RDX trên nền polyethylen wax oxy hóa

Trong bài báo này, một số yếu tố ảnh hưởng đến công nghệ chế tạo thuốc nổ A-IX-1O bằng phương pháp biến tính trong hệ nhũ tương nước có sử dụng sáp polyethylen wax oxy hóa (OPE wax) trong dung môi cyclohexan kết hợp chất hoạt động bề mặt octylphenol ethoxylate (Triton X-100) đã được nghiên cứu. Kết quả nghiên cứu cho thấy rằng, việc thay thế sáp ocxyzin bằng cách sử dụng sáp OPE wax với nồng độ hòa tan trong dung môi cyclohexan không quá 5% kết hợp chất hoạt động bề mặt Triton X-100 ở nồng độ dung dịch thấp không quá 0,1% hexogen (RDX) làm cải thiện khả năng bao phủ của lớp sáp OPE wax lên bề mặt hạt RDX. Kết quả là, các hạt tạo ra có độ mịn, hạt nhỏ đồng đều hơn và không xảy ra hiện tượng vón cục. Thuốc nổ A-IX-1O được chế tạo trong nghiên cứu này thể hiện các đặc tính kỹ thuật tương đương với thuốc nổ A-IX-1O của Nga, nhưng có độ nhạy va đập thấp hơn đáng kể.

Từ khóa: Thuần hóa thuốc nổ; Thuốc nổ A-IX-1O; Polyethylen wax oxy hóa; Chất hoạt động bề mặt Triton X-100.