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MODIFIED PBXN-5 EXPLOSIVE: EFFECT OF IF-WS_2 NANOPARTICLES ON PHLEGMATIZATION OF HMX

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Abstract: This study examines the influence of inorganic fullerene-like tungsten disulfide nanoparticles (IF-WS_2) on the rheological behavior of Viton A solutions and their application in the phlegmatization of octogen (HMX). IF-WS_2 nanoparticles were selected due to their unique quasi-spherical morphology, lubricating properties, thermal and mechanical resistance. Their incorporation into fluoropolymer binders like Viton A is expected to reduce interparticle friction and enhance energy dissipation under mechanical stimuli, thereby decreasing sensitivity to impact and friction. At the same time, IF-WS_2 nanoparticles influence the processing parameters such as viscosity and particle coating efficiency. Two polymer-bonded explosive (PBX) formulations were prepared via aqueous suspension coating: a reference system HMX/Viton A, known as a standardized explosive PBXN-5, and a modified system containing 1 wt.% IF-W_2 dispersed in the polymer solution. The binder was introduced as a solution in ethyl acetate, while nanoparticles were ultrasonically dispersed. Rheological behavior of the solutions were examined by viscosity measurements over time, performed using an MCR 302 rheometer at constant shear rate and temperature. The presence of IF-WS_2 significantly accelerated increase in viscosity, indicating enhanced solvent evaporation and possible nanoparticle–polymer interactions. After drying, PBX granules were characterized by bulk density, sieve analysis of the particle size distribution, microscopic analysis, DSC, and sensitivity to impact and friction. The results suggest that IF-WS_2 nanoparticles influence both processing behavior and final material properties, offering potential for improved control of PBX performance and safety.

Keywords: *HMX, Viton A, PBX, IF-WS_2 nanoparticles, rheology, sensitivity.*

1. INTRODUCTION

Polymer-bonded explosives (PBX) represent an important class of energetic materials designed to combine high explosive performance with improved mechanical properties and reduced sensitivity to accidental initiation. Their structure consists of energetic crystalline particles embedded in a polymeric binder matrix, which provides mechanical integrity, improves handling safety, and protects explosive crystals from external mechanical stimuli. They may be produced by different technologies and using various types of polymers: like granular formulations with thermoplastic binder, intended for pressing, then like cast-cured polymers with elastomeric binder, or like hand-mealable putty-like explosives with highly plasticized partly cured elastomeric binders [1-4]. In case of granular compositions they represent explosive materials in which explosive powder is bound together in a matrix using 5-10wt.% of a synthetic polymer. These PBXs were first developed in 1952 at Los Alamos National Laboratory, as RDX embedded in polystyrene with plasticizer [1, 5]. Later on, during the following decades, formulations were developed using other nitramines, like HMX, PETN, CL-20, or other high explosives, with a wide variety of polymer phlegmatizers, like polycarbonate, PMMA, Teflon, fluoropolymers like Viton, thermoplastic elastomers like Estane, and some energetic polymers [2, 4].

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Among the high explosives used in PBXs, hexogen (RDX) and octogen (HMX) remain most widely employed secondary explosives due to their high detonation velocity, high detonation pressure, and excellent energy output. However, the sensitivity to impact and friction necessitates the application of efficient phlegmatization techniques to improve safety without significantly compromising energetic performance.

Fluoropolymer binders are frequently utilized in PBXs because of their excellent chemical compatibility with nitramines, thermal stability, hydrophobicity, and ability to reduce mechanical sensitivity [4-6]. Viton A, a copolymer based on vinylidene fluoride and hexafluoropropylene, is among the most commonly used fluoropolymer binders in military PBX formulations. One of the best-known examples is PBXN-5, which consists predominantly of HMX particles coated with a small amount of Viton A binder (95/5 wt.%). The performance and safety characteristics of such materials strongly depend on the quality of particle coating, binder distribution, particle size distribution, and the microstructure developed during the coating process [7-9].

In recent years, significant attention has been devoted to the incorporation of nanomaterials into energetic formulations. Various nanoparticles have been investigated as additives capable of modifying mechanical, thermal, and sensitivity characteristics of explosives and propellants. Due to their high specific surface area and unique physicochemical properties, nanoparticles can alter crystal-binder interactions, influence heat transfer mechanisms, modify rheological behavior during processing, and improve resistance to accidental mechanical initiation [10]. The use of nanostructured additives therefore offers new possibilities for tailoring the performance and safety of advanced energetic materials.

Among the available nanomaterials, inorganic fullerene-like tungsten disulfide (IF-WS_2) nanoparticles have attracted considerable scientific and technological interest. Unlike conventional layered WS_2 particles, IF-WS_2 nanoparticles possess a closed-cage, quasi-spherical morphology consisting of multiple concentric WS_2 layers. This unique structure provides exceptional tribological properties, including low friction coefficients, high wear resistance, and efficient energy dissipation under mechanical loading. These nanoparticles exhibit excellent thermal stability, high mechanical strength, and good compatibility with polymer matrices [11-18]. Previous studies have demonstrated their effectiveness as reinforcing and lubricating additives in various polymer composites, where improvements in mechanical and tribological performance have been observed [17-22]. The tribological characteristics of IF-WS_2 nanoparticles suggest that they may also contribute to reducing the sensitivity of energetic materials. Mechanical initiation of PBXs is commonly associated with localized energy concentration and hotspot formation occurring at crystal contacts, defects, or frictional interfaces. By reducing interparticle friction and promoting more uniform stress distribution, IF-WS_2 nanoparticles may mitigate the mechanisms responsible for hotspot generation. In addition, their presence may influence the rheological behavior of polymer binder solutions during PBX preparation, affecting coating efficiency, solvent evaporation, particle agglomeration, and the final microstructure of the explosive composition.

Despite the extensive investigation of IF-WS_2 nanoparticles in tribological and polymer-composite applications, their potential use in polymer-bonded explosives has not been explored or not reported. In particular, little information is available regarding their influence on the rheological properties of fluoropolymer binder systems and their role during the aqueous suspension coating process, which is commonly employed for RDX or HMX phlegmatization. Therefore, the objective of this study was to investigate the effect of IF-WS_2 nanoparticles on the preparation and properties of PBXN-5 explosive. Particular attention was devoted to the rheological behavior of Viton A solutions containing dispersed nanoparticles, as well as to the resulting particle morphology, granulometric characteristics, thermal behavior, and sensitivity to impact and friction.

2. EXPERIMENTAL

2.1. Materials and PBX preparation

The materials used for the preparation of PBX formulations in this study were the following:
- octogen (HMX), supplied by Prva Iskra – Namenska, Barič, Serbia;
- Viton A fluoropolymer binder (DuPont);
- inorganic fullerene-like tungsten disulfide nanoparticles (IF-WS_2) produced by ApNano, NanoLub – Israel;
- ethyl acetate as the solvent for the polymer binder, MERCK; and
- distilled water.

Two HMX-based PBX formulations were prepared. The reference composition corresponded to a conventional PBXN-5 formulation consisting of HMX coated with Viton A binder in ratio 95/5 wt.%. The modified formulation contained the same HMX/Viton A system with the addition of 1 wt.% IF-WS_2 nanoparticles (regarding the amount of Viton A) dispersed within the polymer solution prior to coating. The PBX samples were prepared using the aqueous suspension coating method based on polymer precipitation from solution. HMX crystals were first dispersed in water in a jacketed glass reactor equipped with mechanical stirring and temperature control. Viton A was dissolved in ethyl acetate to obtain a homogeneous polymer solution. For the nanomodified formulation, IF-WS_2 nanoparticles were dispersed in the Viton A solution by ultrasonic treatment before addition to the explosive suspension in order to ensure uniform nanoparticle distribution. The polymer solution was then gradually introduced into the stirred HMX/water suspension. During continuous stirring and controlled heating, ethyl acetate evaporated, causing precipitation of the polymer onto the surface of HMX crystals, producing PBX granules. In the nanomodified system, IF-WS_2 nanoparticles became incorporated within the precipitated

polymer coating.

After the complete solvent evaporation, the coated granules were separated, thoroughly washed with water to remove residual processing impurities, and dried in a laboratory oven at moderate temperature to avoid softening of the polymer binder and agglomeration of the granules. Part of the preparation procedure is schematically shown in Figure 1.

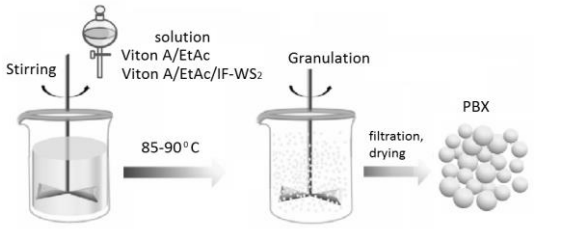


Figure 1. Ilustration of preparation of PBX samples

2.2. Methods

Prior to addition of the HMX suspension in water, small amount of the polymer phlegmatizer solutions in ethyl acetate, with and without the nanoparticles of tungsten disulfide, was taken aside and submitted to examination of viscosity on a rheometer, to monitor the change in apparent viscosity with time caused by the evaporation of ethyl acetate and to evaluate the influence of WS_2 particles on this change. The measurements were carried out using an Anton Paar MCR 302 rotational rheometer equipped with a 35 mm parallel-plate measuring system and P-PTD 220 and H-PTD 220 temperature-control elements. The gap between the plates was set to 1 mm, and the measurement temperature was maintained at 25 °C. Each sample was tested at a constant shear rate of 0.1 s^{-1} for 30 min. The apparent viscosity was continuously recorded as a function of time.

The prepared PBX granules were characterized using a combination of physical, chemical, thermal, and sensitivity analyses. Bulk density was determined by measuring the mass of a known volume of freely poured granules. The morphology and surface characteristics of the coated granules were examined by optical microscopy in order to assess particle shape, coating quality, and the presence of coarse agglomerates. This control of the phlegmatization success was done using LEICA M205A stereo microscope. Particle size distribution was evaluated by dynamic image analysis using DIA 500 Anton Paar device, in liquid mode, with water as a dispersion medium.

Thermal behavior was investigated by differential scanning calorimetry (DSC), using device Nexta DSC200, to determine characteristic phase transitions and decomposition temperatures of the prepared compositions and to assess the influence of IF-WS_2 nanoparticles on the thermal stability of the explosive material. Measurement was done in a temperature interval -80 to 325°C.

The safety characteristics of the PBX formulations were evaluated through standard sensitivity tests. Impact sensitivity was determined using a drop-hammer apparatus Julius Peters by measuring the response of the samples to controlled mechanical impact, using standard steel collars and cylinders made by OZM Research. Friction sensitivity was assessed using a friction tester Julius Peters under standardized loading conditions, using standard porcelain plates and pins produced by OZM Research.

3. RESULTS AND DISCUSION

3.1. Rheological behavior of the polymer solutions

The examined samples of neat and nanomodified Viton A/EA system showed a similar three-stage change in apparent viscosity with time, shown in Figure 2.

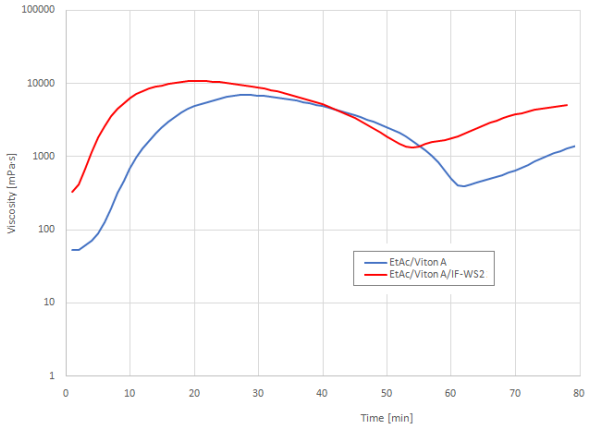


Figure 2. Apparent viscosity as a function of time for Viton A/ethyl acetate solutions with and without WS_2.

The apparent viscosity increased during the first 7-9 min of the measurement until a maximum value was reached. This initial increase was expected due to the progressive evaporation of ethyl acetate, which increased the concentration of Viton A and, in the nanomodified sample, the concentration of solid particles. After reaching the maximum, the apparent viscosity decreased until approximately 18-21 min. A second increase was then observed and continued until the end of the measurement. As it can be seen, a higher peak apparent viscosity was detected for the Viton A/EA/WS_2 sample, which reached 10.77 Pa·s after 7.0 min. Viton A/EA reference sample reached 7.09 Pa·s after 9.3 min. The apparent viscosity decreased to 1.32 Pa·s at 18.0 min for the WS_2-containing sample, and 0.393 Pa·s at 20.7 min for the sample without the nanoparticles. At the end of the measurement, the apparent viscosity increased again to 7.24 Pa·s for the Viton A/EA/IF-WS_2 dispersion, and 2.40 Pa·s for the Viton A/EA system. In the parallel-plate geometry, the sample was exposed to the surrounding atmosphere mainly along the outer edge of the plates. Therefore, solvent evaporation was probably more pronounced in this region, resulting in the formation of a solvent-depleted, polymer-rich layer. Under continuous shear, this layer may have been progressively disrupted or partially detached. Edge effects, including possible wall slip or partial loss of contact between the sample and the measuring plates, may have reduced the measured torque

and caused the observed decrease in apparent viscosity. The subsequent increase was most likely related to the continued evaporation of ethyl acetate and the resulting increase in the concentration of Viton A and solid particles in the remaining sample.

3.2. Morphology of the PBX granules

The appearance of the granules of the reference PBXN-5 and the nanomodified formulation are given in Figure 3. As may be observed, both formulations appear to have expected morphology of the granules, typical for this type of PBX [7-9].

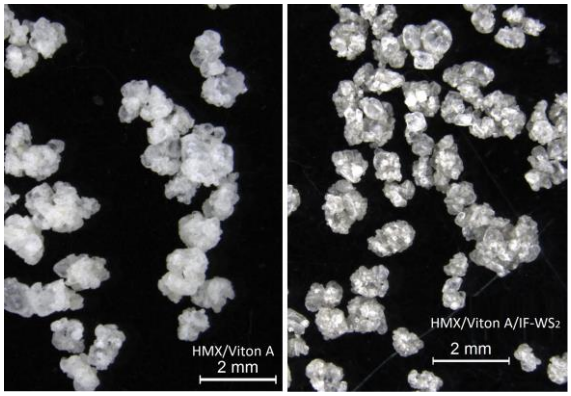


Figure 3. Micrographs of the PBX granules

3.3. FTIR analysis results

FTIR spectra registered for the analyzed PBXs are given in Figure 4.

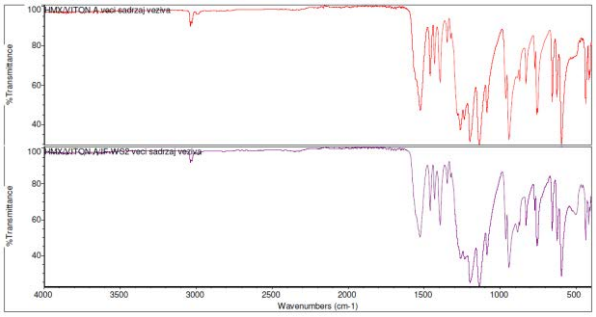


Figure 4. FTIR spectra of the prepared PBXs

It may be observed that there is no significant difference between the registered spectra, so the addition of WS_2 nanoparticles does not have a notable chemical interaction. FTIR spectra of the reference and nano-modified PBXN-5 samples exhibited the same characteristic absorption bands. No new absorption peaks or significant band shifts were observed after incorporation of IF-WS_2 nanoparticles, indicating that the modification did not induce chemical changes in the PBX matrix. Minor variations in band intensities suggest predominantly physical interactions between the nanoparticles and the composite constituents, and to differences in component distribution at the specific point of ATR contact.

3.4. Granulometric size distribution and loose bulk density of the PBX granules

The results of the measurement of the particle size are given in Figure 5. It may be noted that the obtained granules had practicaly same size distribution.

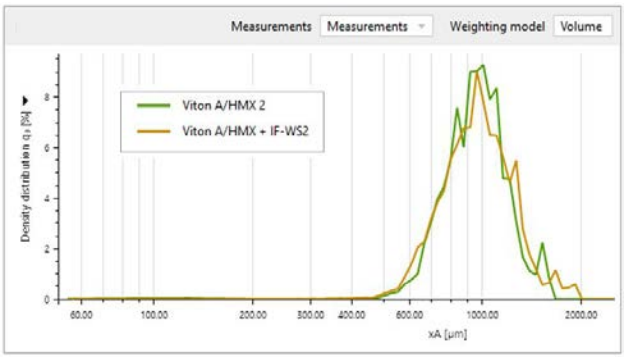


Figure 5. Size distribution of the PBX granules

For the reference PBXN-5 sample, loose bulk density was measured to be 654.1 g/dm^3, and for the WS_2-nanomodified PBXN-5 it was 667.1 g/dm^3. The obtained values are similar to those obtained earlier for PBXN-5 [7-9]. This small difference in the loose bulk density might have explanation in improved packing and reduced void volume caused by the presence of IF-WS_2 nanoparticles in the phlegmatizer [23-26].

3.4. DSC results

DSC thermograms of the HMX/Viton and HMX/Viton/IF-WS_2 formulations exhibited similar thermal behavior over the entire investigated temperature range. The DSC thermograph of the analyzed PBX sample HMX/Viton A is given in Figure 6.

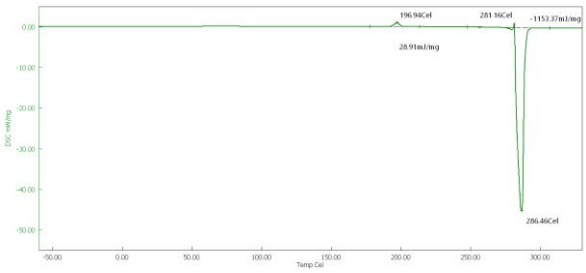


Figure 6. DSC thermograph for PBXN-5

An endothermic event observed at approximately 197-198°C corresponds to the well-known β to δ polymorphic transition of HMX. The transition temperature showed only a minor increase from 196.94 °C for HMX/Viton to 198.22 °C for HMX/Viton/IF-WS_2. At higher temperatures, both formulations displayed a pronounced exothermic decomposition peak centered at approximately 286.5 °C, indicating that the incorporation of IF-WS_2 nanoparticles does not significantly affect the thermal stability of HMX. A small increase in the decomposition peak temperature was observed for the nanomodified formulation (286.54 °C compared to 286.46 °C for the reference sample), although the magnitude of this shift is within the typical uncertainty of DSC measurements. Similarly, the melting endotherm of

HMX, partially overlapped by the onset of decomposition, appeared at slightly higher temperatures in the presence of IF-WS_2 (281.50 °C compared to 281.16 °C). Overall, all characteristic thermal events exhibited a consistent but very small shift toward higher temperatures after the addition of IF-WS_2, suggesting a weak stabilizing effect associated with nanoparticle-matrix interactions. However, additional measurements would be required to confirm the statistical significance of this trend. The slightly higher decomposition enthalpy observed for the formulation with IF-WS_2 may indicate a more complete decomposition process resulting from improved thermal coupling and enhanced interfacial contact within the composite microstructure. A higher decomposition enthalpy was recorded for the IF-WS_2-modified formulation (1263 J g^{-1}) compared to the reference composition (1153 J g^{-1}). However, considering that only a single DSC measurement was performed for each sample and that the analyzed sample masses were approximately 1 mg, this difference should be interpreted cautiously until confirmed by replicate measurements.

A more pronounced effect of IF-WS_2 was observed in the glass transition region of the Viton binder. The part of DSC thermographs of the compared samples where we may observe enlarged part with detectable Tg is given in Figure 7.

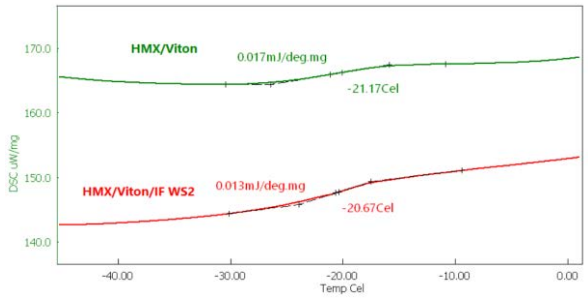


Figure 7. Enlarged DSC thermographs with Tg of Viton A detected in PBXN-5 and PBXN-5/IF-WS_2

in the glass transition region revealed a Tg value of -21.17 °C for the HMX/Viton formulation and -20.67 °C for the HMX/Viton/IF-WS_2 composite. The incorporation of IF-WS_2 nanoparticles therefore resulted in a slight increase of the glass transition temperature by approximately 0.5 °C. Although the shift in Tg is relatively small, a noticeable reduction in the heat capacity increment at the transition was observed. The ΔCp value decreased from 0.017 mJ·deg^{-1}·mg^{-1} for the reference formulation to 0.013 mJ·deg^{-1}·mg^{-1} after the addition of IF-WS_2, corresponding to a reduction of approximately 24%. The negligible change in Tg suggests that IF-WS_2 does not significantly alter the molecular structure of the Viton binder nor induce extensive changes in chain flexibility. However, the reduced ΔCp indicates the presence of restricted polymer segments at the polymer–nanoparticle interface. Such behavior is commonly attributed to physical adsorption of polymer chains on the nanoparticle surface, resulting in the formation of an interfacial region with reduced segmental mobility. The nanoparticles slightly constrain the motion of Viton chains without substantially affecting the overall glass transition temperature. The reduced ΔCp indicates that a fraction of the Viton chains becomes immobilized at the IF-WS_2 surface, suggesting stronger filler-binder interactions and a more constrained interfacial polymer layer.

3.5. Sensitivity to impact and friction

The results of sensitivity tests performed on the prepared granular PBXs are given in Table 1.

Table 1. Sensitivity of the prepared granular PBXs

Sample	Impact sensitivity	Friction sensitivity
PBXN-5	3.14 J	324 N
PBXN-5/IF-WS_2	2.94 J	240 N
PBXN-5/graphite	2.76 J	268 N

Contrary to the expected desensitizing effect of solid lubricant nanoparticles, IF-WS_2 slightly increased the mechanical sensitivity of PBXN-5. This behavior may be attributed to the introduction of additional interfacial regions, local stress concentration sites within the PBX microstructure and to the presence of hard nanoscale particles. This might have helped formation of hot-spot under mechanical stimuli [6, 23]. This means that in order to achieve the desired effects, different approach of incorporation of the nanostructures should be applied, so the further research will consider possibility of applying a thin layer of the solid lubricating particles ontop of the PBX granules.

CONCLUSIONS

Nanomodified PBXN-5 was prepared and characterized. The incorporation of 1 wt.% IF-WS_2 nanoparticles into the Viton A binder influenced the processing behavior and properties of PBXN-5 explosive. Rheological measurements showed a significant increase in apparent viscosity of the Viton A/ethyl acetate solution with added nanoparticles during solvent evaporation, indicating interactions between the nanoparticles and the polymer solution that may affect coating efficiency and granule formation. FTIR analyses confirmed that the addition of IF-WS_2 did not induce chemical changes in the PBX matrix. DSC demonstrated that the presence of the nanoparticles slightly altered PBX`s thermal stability, with a slight increase in Tg and reduction in ΔCp. The nanomodified formulation exhibited morphology comparable to the reference PBXN-5. However, a slight increase in mechanical sensitivity was observed, which will direct further research towards different technology of incorporation of IF-WS_2 nanoparticles in formulation of PBXs.

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