Preparation of insensitive composites based on penta erythritol tetra nitrate particles coated with carbon black- Triton X114 by a solvent/non-solvent process via Taguchi design optimization

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Abstract

One way to reduce sensitivity and also to add special properties to explosives is to perform coating that depends on either the coating agent type or the usage process. In this work, the insensitive composite of penta erythritol tetra nitrate (PETN) was prepared with carbon black (CB) and Triton X-114 (TX114) by a solvent/non-solvent method. Taguchi experimental design (orthogonal array, L9) with using the impact sensitivity (H_{50}) as a response was applied for the process optimization. Effects of the CB mass fraction, solvent flowrate, surfactant type and surfactant concentration were evaluated and the results were quantified by the analysis of variance (ANOVA). The ANOVA analysis predicted that the best H_{50} was 67.4 ± 1.5 cm for the optimum synthesis conditions of 5.0 wt% CB, 1 mL min^{-1} flowrate, and TX114 as a surfactant at a concentration of 2.0×10^{-3} mol L^{-1}. The experimentally determined value of H_{50} was 68.0 ± 0.5 cm, which is in good agreement with the predicted value. Finally, thermal analysis and vacuum stability test were applied to the synthesized composite indicating that CB and TX114 are thermally adaptable and chemically compatible with PETN.

Keywords: Desensitized PETN; Taguchi design; impact sensitivity; thermal kinetic; coating process

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1. INTRODUCTION

Explosive materials such as penta erythritol tetra nitrate (PETN) and glyceryl trinitrate (GTN) constitute an important group of high energy materials that are widely used for military and civilian applications [1]. Safety, mechanical and thermal properties are important features that limit the use of these compounds in many areas. Nitrate ester compounds are highly sensitive to external stimuli (such as impact, friction, and thermal shock), and their use without desensitization can lead to tragic explosions during the production, storage and also transportation of these substances. PETN is a powerful explosive which exhibits a considerable velocity on detonation (VOD = 8310 m s^{-1} at the density of d = 1.77 g cm^{-3}). It is the most stable and least reactive of the energetic nitrate esters. The relatively high sensitivity of PETN to friction and impact means that it is usually desensitized with phlegmatizers like wax or synthetic polymers to form plastic bonded explosives (PBXs) like datasheet and Semtex. A cast mixture of PETN and TNT in equal proportions is known as pentolite. It has seen wide use as a high explosive in detonators, detonation cords and booster charges for military, road construction, mining, demolition and civilian industries [2].

Therefore a lot of research has been performed to increase safety of these compounds and so to decrease their sensitivity. Many reports have been provided about desensitization of explosives that focused on crystal size, shape, and removal of impurities and defects *via* re-crystallization as well as on polymer-bound explosives [3-7]. In most cases, along with reduced sensitivity, the energy stored in the coated sample will be decreased. Therefore, finding the optimal balance between the minimum sensitivity and the maximum energy content of an explosive is a major problem in the design and production of military ammunition [8].

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In recent years, coating pretreatments such as micro-encapsulation [9], emulsion solvent evaporation [10], crystallization [11], spray drying [12] and solvent/non-solvent (SNS) [13-15] methods were widely used for desensitization of explosives compounds. In this manner, addition of materials involving carbon such as fullerenes, carbon nanotubes, graphite and graphene oxide have been studied [13,16-19].

Due to simple application, a single pot process, lower cost, high efficiency, simple equipment and higher thermal stability of explosives during combustion, SNS techniques are preferable among surface coating methods [13].

Among factors affecting the coating efficiency of a SNS process, the solvent nature, weight ratio of the coating agent to the explosive, time of non-solvent addition and the mixing rate are the most important [13]. With the proper change of each of the parameters, a composite with special physicochemical and safety properties will be produced that can be used for a particular purpose.

One of the many techniques for optimization of the key factors is the Taguchi method [20]. It can identify and organize system interactions within experimental data for the analysis leading to an optimal design [21,22]. Moreover, it is proven that this method can be applied to solve a variety of problems including continuous, discrete and qualitative design variables [23,24]

Taguchi method classifies the analyzed factors as controllable and noise factors. Noise factors are variables that influence response of the process but cannot be controlled economically. They are not maintained at particular levels during the process period for the expected performance at optimum conditions with the least variations [25]. These factors are usually the first sources of variation and due to their control difficulties cannot be considered [26,27].

In this research, a desensitized PETN powder was prepared with the use of carbon black (CB) by a solvent/non-solvent method in a media containing Triton X-114 (TX114) via Taguchi statistical design (orthogonal array, 3^4) method. In this regard, the composite impact sensitivities were evaluated regarding different factors including the CB mass fraction, solvent flow rate, surfactant type and the surfactant concentration.

2. EXPERIMENTAL

2. 1. Chemicals

Carbon black (CB) was obtained from Main chem. (Fujian, China) with a particle size range of 1-6 μm. Highly pure PETN (m.p.: 414.55 K, crystal density: 1.77 g cm^{-3}) was prepared by defense industry (Esfahan, Iran). Sodium dodecyl sulphate (SDS), Triton X-114 (TX114) and N-cetyl-trimethyl ammonium bromide (CTAB) surfactants of analytical grades were purchased from Across (New Jersey, USA). Ultra-pure water was obtained by a Millipore system (Bedford, UK) and the other chemicals were provided from Merck (Darmstadt, Germany).

2. 2. Equipments

Fourier transform infrared (FT-IR) spectra were obtained by using a Nicolet 800 (Thermo Electron Corporation, USA). Scanning electron microscopy with energy dispersive X-ray analysis (Vega3Tscan, TESCAN ORSAY Company, Czech Republic) were used for determination of morphology and size of particles and elemental analysis. Thermal studies were performed by a Netzsch DSC14 Polyma instrument (NETZSCH Company, Germany) applying the heating rate of 5 $^oC\ min^{-1}$ under nitrogen atmosphere. Impact sensitivities were determined by a Julius Peters Apparatus (BAM Fall Hammer Company, Germany) using a BAM method (according to STANAG 4489 standard). According to this method, a certain amount of the energetic material is placed between two steel plates and hit by a hammer (1 kg) which falls from different heights. For each fall height, six replicate runs are performed, and the initiation energy amount of the explosive is calculated. Impact sensitivity can be expressed as H_{50} (drop height corresponding to 50 % probability of initiation). Therefore, the highest H_{50} is desirable. Chemical compatibility measurements were carried out using a vacuum stability test according to STANAG 4556 standard. All data handling for Taguchi method was performed by using MiniTab-17 software (MiniTab Corporation, USA).

2. 3. Procedure

A specific weight of CB was added to a 200 mL beaker containing 100 mL of an aqueous surfactant solution (according to Table 1) and the mixture was subjected to ultrasonic waves (100 W) at 313.15 K for 30 minutes. A PETN solution in acetone at the concentration of 0.12 mol Lit^{-1} was then added drop wise to the mixture while stirring at 333.15 K. After ca. 80 % of the solvent was evaporated, the remaining solution was filtrated. The precipitate was then washed 3 times with 5 mL of water and 2 times with ethanol. The precipitant was dried at 333.15 K for 24 hours in an oven and then its impact sensitivity was measured by the sensitivity apparatus. A schematic diagram of the preparation procedure is shown in Figure 1. In order to obtain efficient coating of PETN powder by CB particles, parameters of the coating process were optimized by using the Taguchi statistical method. This experimental design based on OA_9 (3^4) was performed by changing four variables (*i.e.* CB mass fraction, solvent flow rate, surfactant type and surfactant concentration) at 3 levels that are summarized in Table 1.

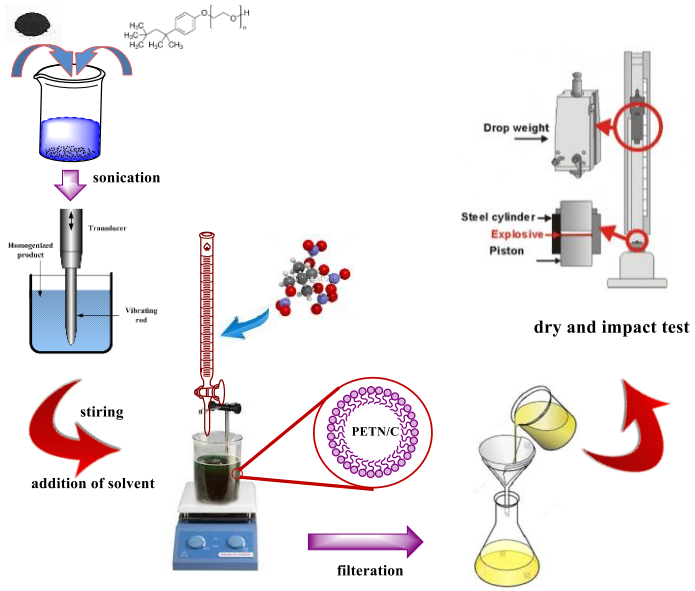

Figure 1. *A CB amount was transferred to an aqueous surfactant solution and after ultrasonication, the PETN was added to the mixture gradually. After filtration and washing, the precipitant was dried thoroughly and its impact sensitivity was measured*

Table 1. *Arrangement of the factors and levels of the experiments (OA_9 matrix)*

Trial no.	CB mass fraction, Wt%	Solvent flowrate, mL min^{-1}	Surfactant type	Surfactant Concentration, mol Lit^{-1}
1	1	1	SDS	1.0×10^{-3}
2	1	2	TX-114	2.0×10^{-3}
3	1	3	CTAB	4.0×10^{-3}
4	3	1	TX-114	4.0×10^{-3}
5	3	2	CTAB	1.0×10^{-3}
6	3	3	SDS	2.0×10^{-3}
7	5	1	CTAB	2.0×10^{-3}
8	5	2	SDS	4.0×10^{-3}
9	5	3	TX-114	1.0×10^{-3}

3. RESULTS AND DISCUSSION

3. 1. FT-IR spectroscopy

Infrared spectroscopy was used for the composition investigation of the produced composite. Figure 2 shows FT-IR spectra of the PETN, TX114 and the synthesized composite samples. In Figure 2a, related to the PETN spectrum, main

absorption peaks occurred at 2932, 1646, 1269, 1000 and 851 cm^{-1}, which are corresponding to stretching vibrations of CH_2, NO_2(symmetry), NO_2 (asymmetry), CO and NO groups, respectively.

The TX114 spectrum (Fig. 2b) has a broad absorption peak of stretching vibrations for hydroxyl groups at 3347--3459 cm^{-1}, two shoulder peaks of symmetric and asymmetric stretching vibrations for CH_2 groups at 2870 and 2953 cm^{-1}. The intense peaks at 1610 and 1512 cm^{-1} related to stretching vibrations of the benzenoid group can be observed together with the asymmetric stretching of associated aromatic ether at 1247 and 1119 cm^{-1} [28].

As shown in Figure 2c, related to the composite spectrum, presence of TX114 in the coated PETN was confirmed due to the existence of a broad peak arising from the OH functional group at 3347-3459 cm^{-1}. CH_2 peaks arising at 2901 and 2919 cm^{-1} were altered both in shape and intensity as compared to the initial pattern of CH_2 groups in the PETN spectrum. Also peaks at 1119 and 1512 cm^{-1} referring to the benzenoid and aromatic ether groups are visible too.

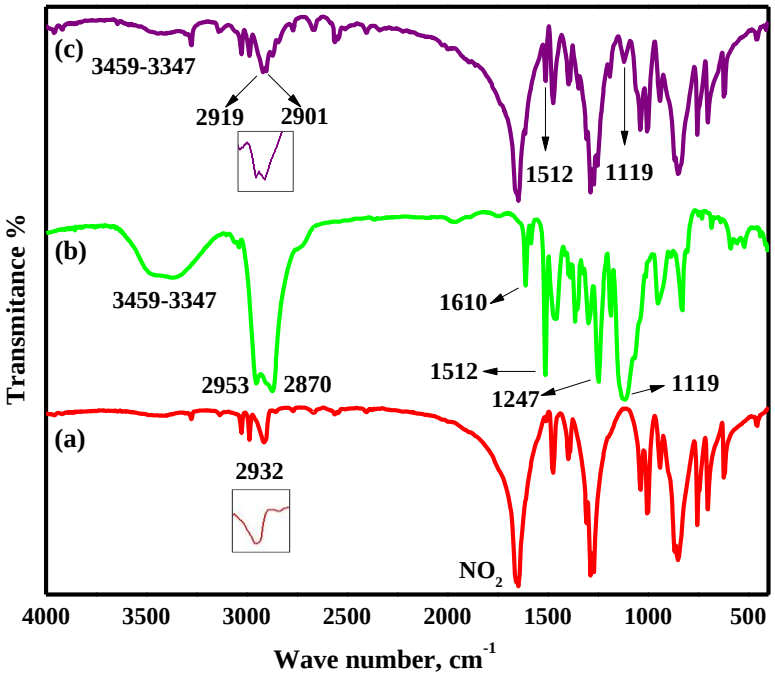

Figure 2. *FT-IR spectra of: (a) PETN; (b) Triton X-114 and (c) PETN\Carbon black\Triton X-114 composite.*

3. 2. Morphological and Elemental Analyses

Morphology, elemental analysis and particle size distributions of PETN and CB powders as well as the synthesized composite were analyzed by using a SEM-EDX system. The results are shown in Figure 3 (a, b and c, respectively).

Figure 3a-1 presents the SEM image of pure PETN showing complex tetragonally-shaped crystals. EDX and carbon mapping analysis (Fig. 3a-2) shows that PETN contains carbon, nitrogen and oxygen at a homogenous dispersion.

The SEM image of CB (Fig. 3b-1) shows amorphous particles with a size range of 1-5 µm. Elemental analysis (Fig. 3b-2) shows partial presence of oxygen that is probably due to slight oxidation of the CB surface.

The SEM micrograph of the prepared composite (Fig. 3c-1) indicates CB particles along with TX114 adsorbed on the surface of PETN crystals. Analysis of the EDX spectrum (n=20) and carbon mapping of the composite compared with that of the pure PETN indicate an increase of carbon content in the synthesized composite that is due to adsorption of CB and TX114 on its surface.

The coating phenomena are explained interactions of hydroxyl groups of the non-ionic surfactant TX114 in the solution with nitro groups present in the PETN structure creating hydrogen bonding [29]. In the present study, it could be hypothesized that the TX114 hydrophilic tails are adsorbed on CB particles, while forming a similar network on the PETN surface (Fig. 4).

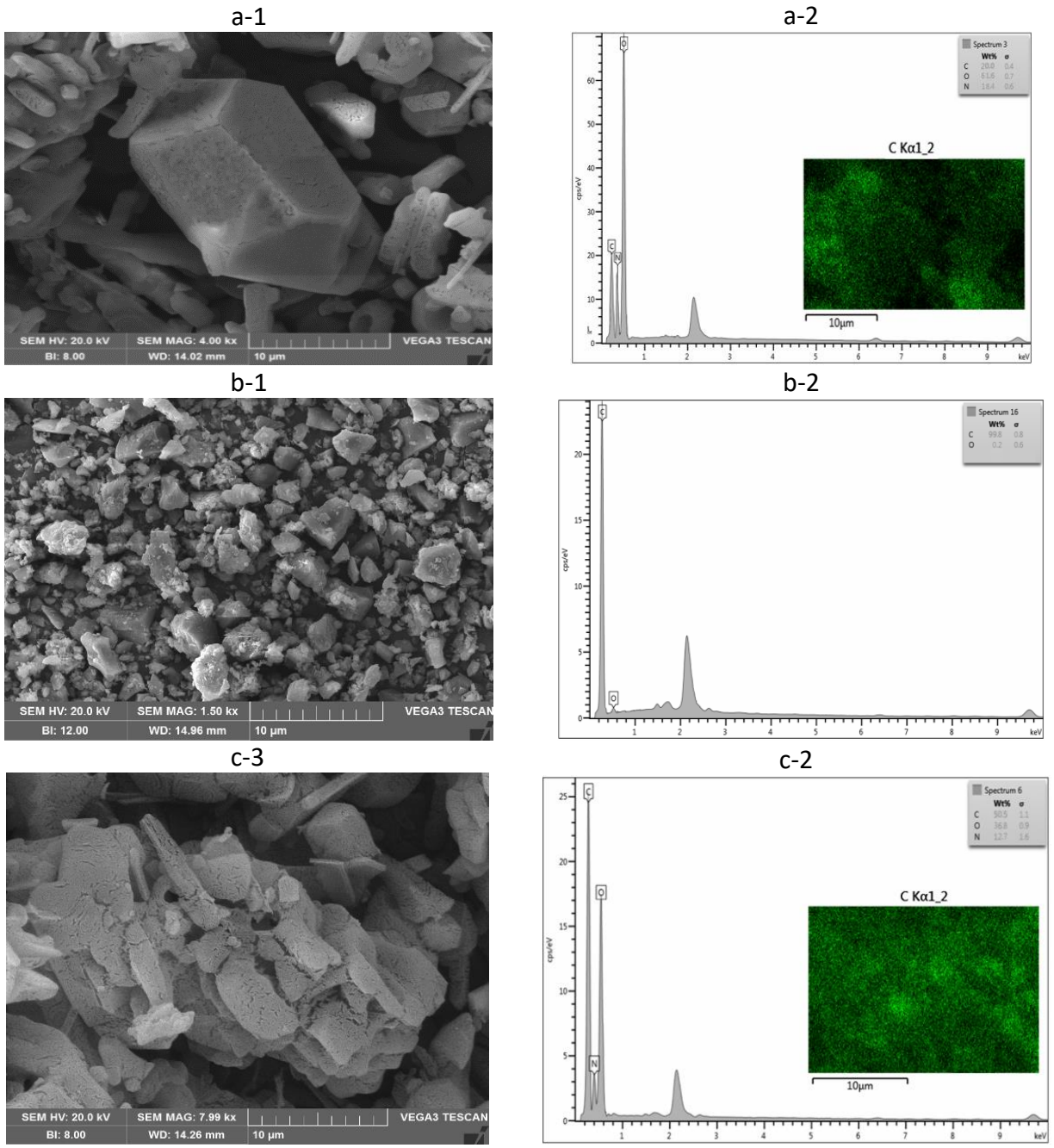

Figure 3. *SEM-EDX-Map analysis of (a) PETN, (b) carbon black and (c) synthesized composite: 1 designates SEM micrographs (scale bar = 10 µm), 2 designates graphs of EDS analysis, insets show carbon maps (scale bar = 10 µm).*

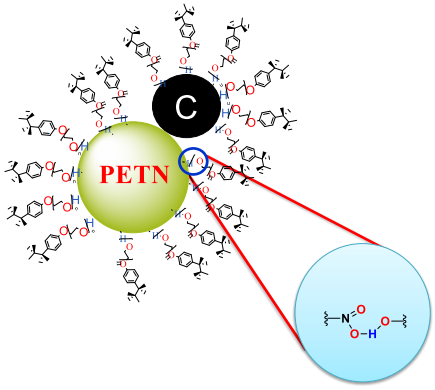

Figure 4. Proposed interactions of a PETN particle with Triton X-114 and carbon black

3. 3. Impact sensitivity and experimental design

Sensitivity is a key property for the safety of explosive materials and has a significant effect on packaging, storage, transportation and application of these materials [30]. Therefore, an impact sensitivity test for manufactured explosives is generally considered. Common optimization procedures for stabilizing a multistage process are sequential and simultaneous methods [31,32]. Sequential methods (each time optimization), are suitable for few response surface designs. These methods show also some difficulties such as slow convergence and requirements for a large number of experiments for a complex response surface with high dimensionality [33].

In simultaneous optimization methods such as mixture designs [34] and factorial designs [35], the mentioned problems are avoided. In these methods after an initial experimental plan, the experimental results are collected and the optimum conditions would be determined by constructing a response surface or by an extrapolated graph. Mixture designs are perfect for experiments in which the response is related to proportions of ingredients in a mixture rather than their values, while factorial designs are used for cases with other variables. A clear weakness of the latter methods is in cases when the number of variables is increased, the number of required experimental trials increases geometrically. Therefore, in this case, implementation of these trials is not feasible and fast. This problem can be reduced by using fractional factorial experimental designs, such as Plackett-Burman schemes or orthogonal array designs (OAD). In comparison to previously developed two-level designs, OAD has three-level designs providing more precise information.

In designing experiments, Taguchi OAD represents the least fractional factorials and is used for the most experimental designs. The number of possible designs, N, in a full factorial design is as follows:

$$N = L^{m} \quad (1)$$

where L is the number of levels for each factor and m is the number of factors. Thus, if the qualities of a given product depend on four factors (A, B, C, and D) and each factor is to be tested at three levels, a full factorial experiment would require 3^4 (81) runs. However, most of these runs do not provide significantly useful information requiring too long time along with high costs. These problems can be solved by orthogonal array design with only 9 beneficial runs (L9) [25, 36].

In this research, four factors (CB mass fraction, solvent flowrate, surfactant type and surfactant concentration) at three levels were considered and Taguchi method was applied for H_{50} studies of the prepared composites. H_{50} values are summarized in Table 2. As shown, the maximum and minimum H_{50} values were obtained in experiments no. 9 and no. 1, respectively. Mean values and effects of the applied factors (along with the mean of means) on H_{50} are shown in Figure 5, which reveals how the H_{50} value of the optimal composite will change when each level of a factor is altered.

Table 2. Experimental results of the selected variables on the impact sensitivity (H_{50})

Trial no.	1	2	3	4	5	6	7	8	9
Impact sensitivity (H_{50}), cm	42.0	53.0	44.0	57.0	60.0	52.0	65.0	50.0	66.0

[a]For six replicate measurements with a standard deviation= ± 0.5 cm

As shown in Figure 5-a, the increase in the CB content induces a significant increase in the H_{50} value, corresponding to a considerable decrease of the composite sensitivity to impact. The results show that the sensitivity of synthesized composites is reduced as compared to the pure PETN (experimentally determined value H_{50} = 35.0 cm) and the sample is more stable. This desensitizing effect may be related to the presence of CB as a neutral component on the composite surface, which by absorbing heat generated in the impact sensitivity test, prevents rising of the PETN temperature, which results in the increased H_{50} value.

Analysis of Figure 5-b indicates that the solvent flow rate has a small effect on the impact sensitivity. The increase of the solvent flow rate over 1 mL min^{-1} has a negligible effect on the impact sensitivity. In general, decreasing the solvent flow rate results in the formation of larger PETN crystals and thus, the interactions between functional groups on the crystalline surface will be increased. This phenomenon was not seen in this coating process as implied by negligible effects of the solvent flow rate on impact sensitivity.

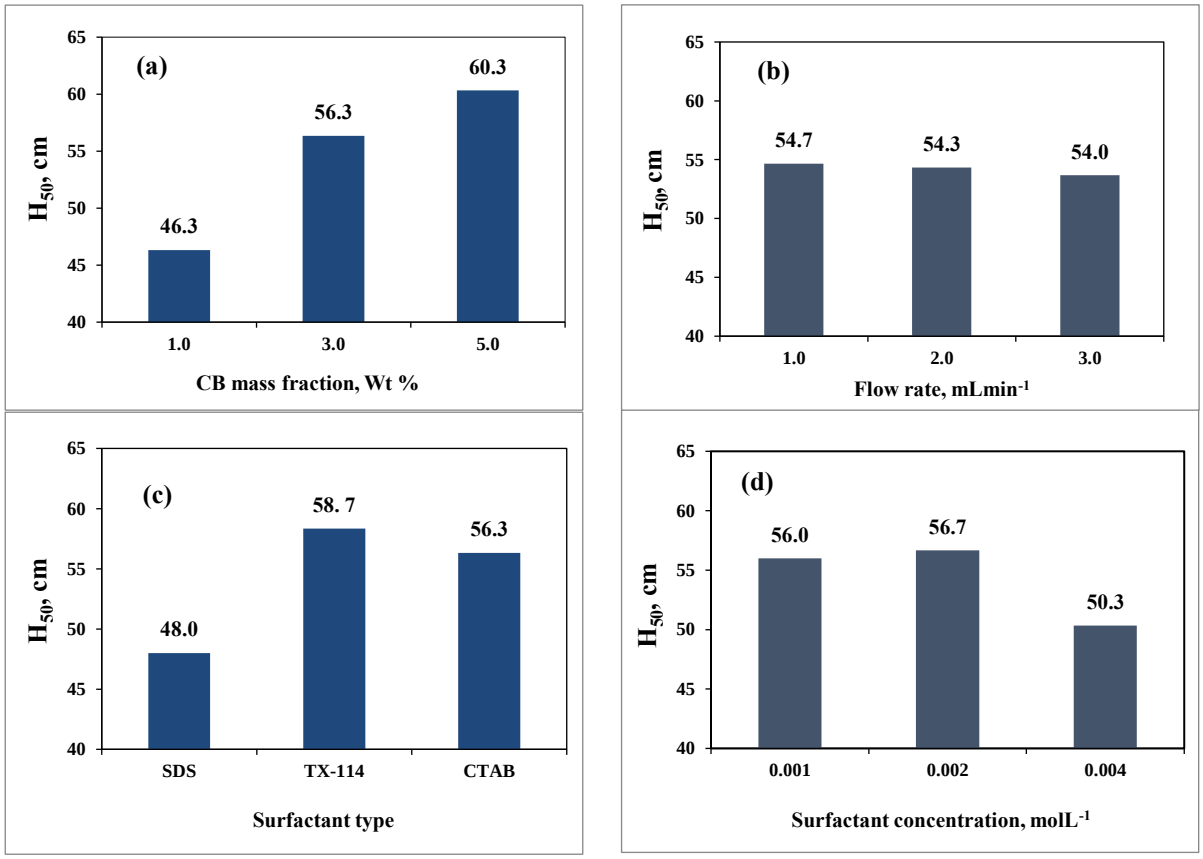

Figure 5. *Effects of optimization parameters (presented are the mean values of means) on impact sensitivity (H_{50}). (a): CB mass fraction, (b): solvent flowrate, (c): surfactant type, (d): surfactant concentration*

Effects of the surfactant type on impact sensitivity of the coated PETN were studied by using three different surfactants: SDS, TX114 and CTAB. Analysis of results of the Taguchi method presented in Figure 5-c show that TX114 exhibits the best influence on H_{50}. This substance is a non-ionic surfactant that contains hydroxyl and benzenoid groups in its structure. These groups probably act similarly to an amphoteric system or a multifunctional chain that interacts with both PETN and CB surfaces. Therefore, the coating process is accomplished to a higher degree leading to the increase in H_{50} values. This effect is not seen in applications of either SDS or CTAB due to their ionic behavior only at one side of a molecule.

Figure 5-d shows that the increase in the surfactant concentration from 1×10^{-3} to 2×10^{-3} mol L^{-1} led to the increase in H_{50} while further increase in the concentration led to a decrease in the H_{50} value. The reason is that the PETN, in addition of CB, is coated by TX114 as another insensitive compound. So, when external hammer force was hit on the sensitized composite, TX114 acts as a lubricant buffer too [37]. At the lower surfactant concentration, H_{50} decreased due to insufficient coating or lower interactions of CB with PETN particles. At the higher concentration than optimal, H_{50} decreased again due to agglomeration of CB particles in the nonsolvent that prevented uniform and complete coating of PETN particles.

Analysis of variance (ANOVA) was applied to evaluate statistical significance of the effects of each factor on H_{50}. According to numerical values of the F-statistic, the value for solvent flowrate is lower than the critical value (at 90 % confidence level) and, thus this factor has to be pooled [38]. Table 3 was obtained after pooling the data for one time. According to the obtained results, the CB mass fraction with participation of 54.24 % has the highest influence on the impact sensitivity. With respect to the obtained data, the optimum conditions for the main factors are sequentially as: the CB mass fraction= 5 wt%, solvent flowrate = 1.0 mL min^{-1}, surfactant type = TX114, surfactant concentration = 2.0×10^{-3} mol L^{-1}.

Table 3. ANOVA results for the Impact sensitivity of the composite by an OA_9 matrix with H_{50} (cm)

Factor	Code	DOF	S	V	Pooled[a]			
					DOF	S´	F´	P´, %
CB mass fraction, wt %	*A*	2	312.000	156.000	2	311.340	468.00	54.24
Solvent flowrate, mL min^{-1}	*B*	2	0.667	0.333	-	-	-	-
Surfactant type	*C*	2	188.667	94.333	2	188.001	283.00	32.75
Surfactant concentration, mol L^{-1}	*D*	2	72.667	36.333	2	72.000	109.00	12.54
Error		0	-	-	2	0.667	-	0.47

DOF: degree of freedom, S: standard deviation, V: variance, S´: standard deviation after pooling, F´: calculated value for the F-test; P´: participation of each factor on the result after pooling.
[a]The critical value was at 90 % confidence level; pooled error results from pooling the insignificant effects.

As a general rule, the optimum performance (here, for impact sensitivity test with highest H_{50}) could be calculated by the equation:

$$Y_{\text{opt}} = \frac{T}{N} + \left(A_3 - \frac{T}{N}\right) + \left(B_1 - \frac{T}{N}\right) + \left(C_2 - \frac{T}{N}\right) + \left(D_2 - \frac{T}{N}\right) \quad (2)$$

where Y_{opt} (H_{50} at the optimum conditions) is equal to the *T*/*N* (ratio of the grand total of all results to the total number of all experiments) plus the contributions of A_3 (the CB mass fraction at level 3, 5 wt %), B_1 (the solvent flow rate at level 1, 1.0 mL min^{-1}), C_2 (the surfactant type at level 2, TX114) and D_2 (the surfactant concentration at level 2, 2.0×10^{-3} mol L^{-1}). The procedure for computation of the confidence interval (CI) of the optimum performance is explained following the equation:

$$\text{CI} = \pm\sqrt{\frac{F_\alpha(f_1, f_2)V_\text{e}}{n_\text{e}}} \quad (3)$$

where, F_α (f_1, f_2) is the critical value for *F* at degrees of freedom (DOF) f_1 and f_2 at the significance confidence level (in this work α= 90 %). f_1 is the DOF of the mean (which always equals to 1), f_2 = DOF of the error term, V_e is the variance of error term (determined by ANOVA), n_e is defined as the effective number of replications, and expressed by n_e = number of trials/(f_1 + DOF of all factors applied in the estimation of optimum results). Statistical calculations for prediction the result and CI at optimum conditions revealed that the H_{50} of the composite will be 67.3 ± 1.5 cm.

For validation of the optimal conditions obtained by the Taguchi method, the impact sensitivity test was applied for the optimal composite. The H_{50} for optimum composite was measured as 68 ± 0.5 cm. This result is in the range of the confidence interval determined above and is acceptable for this work. As a result, by using this coating pretreatment, the H_{50} of pure PETN was increased from 35.0 to 68.0 cm.

3. 4. Thermal analysis

The thermal performance of high-energy materials is considered as a key property and it affects the preparation process, storage and transportation. Therefore, the study of thermal behavior (including stability, sensitivity and energy content) and thermo-decomposition mechanism of PETN or its composite is essential [39,40]. Thermal stabilities of the pure PETN and the optimal composite were investigated by differential scanning calorimetry analysis (DSC) and the thermograms are shown in Figure 6.

Two peaks are observed in DSC curves of the PETN sample and the prepared composite. The first sharp endothermic peak at 413-414 K corresponds to the melting points while the second broad exothermic peak seen in the range of 455-485 K relates to the thermal decomposition of pure PETN [41]. Thus, significant changes of the melting and decomposition temperatures of the synthesized composite as compared to pure PETN were not observed and therefore this negligible difference indicated negligible influences of CB and TX114 on thermal properties of the composite.

DSC behaviors of PETN and composite samples were studied by non-isothermal measurements under different heating rates of 5, 10, 15 and 20 K min^{-1} by the Kissinger method in order to determine thermal decomposition kinetic

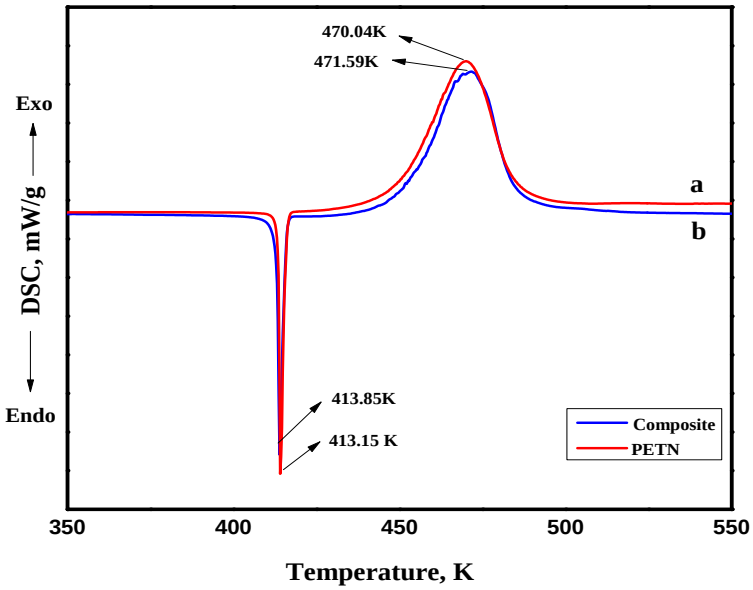

Figure 6. *DSC thermograms of the pure PETN (a) and the synthesized composite (b)*

parameters [40, 42]. As it can be seen in Figure 7 (a-1 and b-1) with increasing the heating rates, the temperature of the exothermic peak increases sequentially. The Kissinger equation is presented as follows:

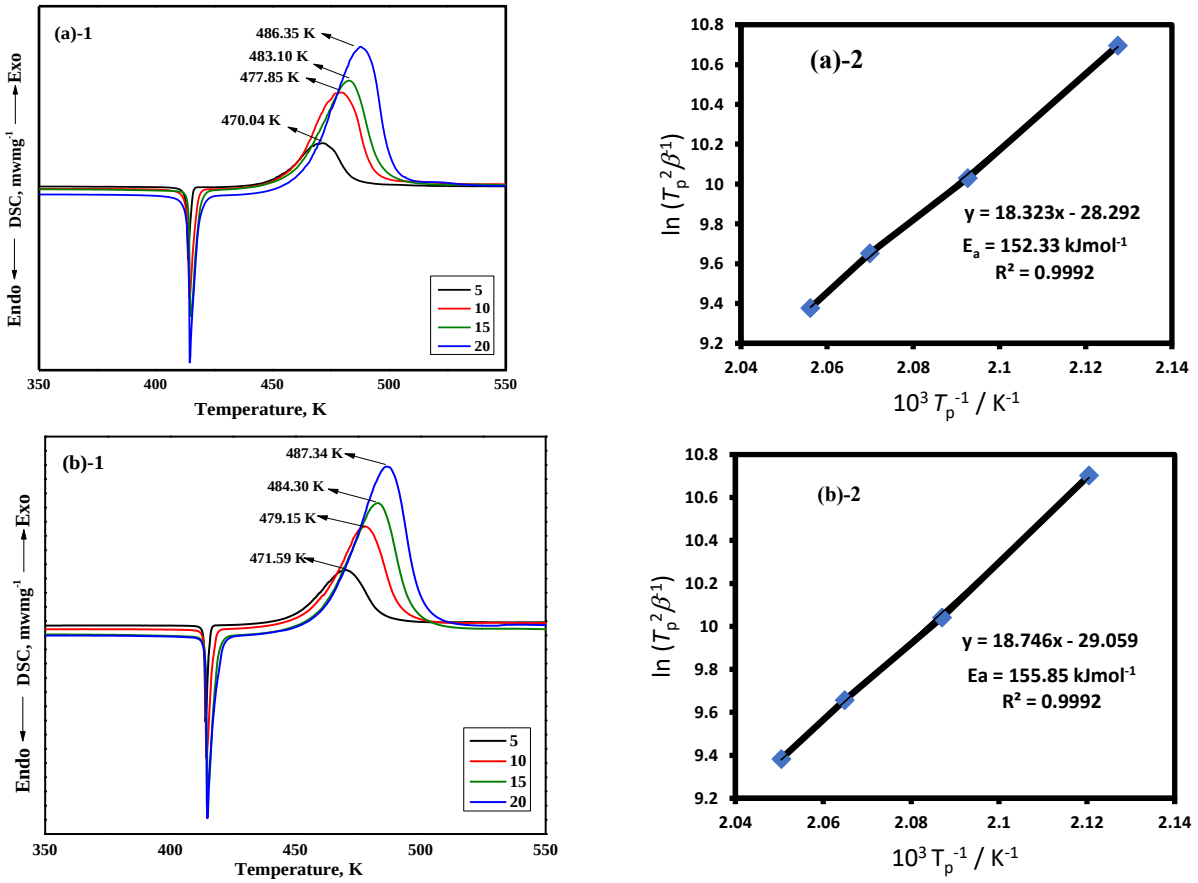

Figure 7. *DSC curves with Kissinger plots of PETN (a1-2) and the sensitized composite (b1-2)*

$$\ln\left(\frac{T_p^2}{\beta}\right) = \frac{E_a}{RT_p} - \ln\left(\frac{AR}{E_a}\right) \quad (4)$$

where T_p is the temperature of the exothermic peak, K; β is the heating rate, K min^{-1}; E_a is the activation energy, J·mol^{-1}; R is the ideal gas constant, 8.314 J·mol^{-1} K^{-1} and A / min^{-1} is the pre-exponential factor. According to the Eq. (4) when ln (T_p^2 β^{-1}) values are plotted against T_p^{-1} values, a straight line is obtained that E_a/R and ln (AR/E_a) factors are the slope and intercept of the line, respectively. The calculated activation energies of PETN and the composite are 152.33 and 155.85 kJ mol^{-1}, respectively (Figs 7-a2 and 7-b2). The small change in the activation energy between the pure PETN and the sensitized composite showed that the added CB did not alter the decomposition mechanism of the pure PETN.

3. 5. Vacuum stability test

One of several ways for measuring the chemical compatibility of explosives is a vacuum stability test (VST). In this technique, the volume of released gas from an energetic compound is measured under a defined temperature at a specific time followed by comparison of the obtained result with the standard value. If the resulting quantity is lower than the standard value, the examined compound is stable. According to the STANAG 4556 standard, the required temperature for PETN is 393.15 K over 20 hours. The VST results for pure PETN and the prepared composite are shown in Table 4. The obtained data indicate chemical compatibility of CB and TX114 with PETN.

Table 4. Data results of vacuum stability test for PETN and the prepared optimal composite

Sample	Volume of evolved gas, mL g^{-1}	Volume of standard evolved gas, mL g^{-1}
PETN	0.18	2.0
Optimal composite	0.19	2.0

4. CONCLUSION

In this research, an insensitive composite of PETN\CB\TX114 was prepared via a solvent/nonsolvent method. FT-IR, SEM, EDX and carbon mapping analyses indicated that the PETN was coated by CB along with TX114. The Taguchi design method was applied to optimize experimental conditions of the preparation process. In this manner, four factors (CB mass fraction, solvent flowrate, surfactant type and surfactant concentration) at three levels were considered while impact sensitivity (H_{50}) responses were used for the experimental design optimization. According to the Taguchi method, optimal parameters for the composite synthesis were 5 wt% CB mass fraction (P= 54.24 %) and TX114 as a surfactant type (P = 32.75 %) at the concentration of 2×10^{-3} mol L^{-1} (P= 12.54 %). The ANOVA analysis showed that the effect of the solvent flowrate was negligible. Sensitivities to impact under optimum conditions predicted by the Taguchi design (H_{50}=67.4 ± 1.5 cm) and experimentally determined (H_{50}= 68.0 ± 0.5 cm) were in satisfactory agreement. By using the proposed synthesis method the H_{50} was increased from 35.0 cm for the pure PETN to 68.0 cm for the optimal composite. Complementary studies of the thermal analysis (decomposition kinetics) and vacuum stability tests showed thermal properties and chemical compatibility of PETN, CB and TX114 as ingredients in the produced composite. The findings obtained in the present work reveal that the proposed method is suitable for PETN coating with a reliable sensitivity decrease along with negligible energy reduction.

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SAŽETAK

Dobijanje eksploziva smanjene osetljivosti na bazi čestica pentaeritrol-tetranitrata prevučenih česticama čađi i površinski aktivnom materijom Triton X114 procesom sa i bez rastvarača uz primenu optimizacije Taguči metodom

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Oblaganje predstavlja jedan od načina za smanjenje osetljivosti eksploziva, kao i za postizanje dodatnih specifičnih karakteristika što zavisi od samog tipa prevlake ili od primene. U ovom radu, dobijen je neosetljiv kompozit na bazi pentaeritrol-tetrantrata (PETN) sa česticama čađi i površinski aktivnom materijom Triton X-114 (TX114) primenom metode sa i bez rastvarača. Za optimizaciju procesa primenjen je Taguči eksperimentalni dizajn (ortogonalno polje, L9) pri čemu je osetljivost na udar (H_{50}) predstavljala odzivnu veličinu. Ispitani su uticaji masenog udela čađi, protoka rastvarača i tipa i koncentracije emulgatora, a rezultati su analizirani primenom analize varijanse (ANOVA). Ovom analizom je predviđeno da će se dobiti najbolja osetljivost na udar od 67,4 ± 1,5 cm pri sledećim uslovima sinteze: 5,0 mas.% čađi, protok od 1 ml min^{-1} i TX114 kao emulgator u koncentraciji od $2{,}0\times10^{-3}$ mol dm^{-3}. Za kompozit dobijen pri ovim optimalnim uslovima sinteze eksperimentalno je određena vrednost H_{50} od 68,0 ± 0,5 cm što je u saglasnosti sa predviđenom vrednošću. Najzad, termalna analiza i test stabilnosti u vakuumu primenjeni na kompozit sintetisan pri optimalnim uslovima su pokazali da su čađ i TX114 termički i hemijski kompatibilni sa PETN.

Ključne reči: Neosetljiv pentaeritrol-tetranitrat, Taguči dizajn, osetljivost na udar, termalna kinetička analiza, prevlake