

Characterization and Performance Evaluation of Recrystallised Micro PETN

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ABSTRACT

This manuscript shows the recrystallization of micro Pentaerythritol tetranitrate (mcPETN) through solvent anti solvent technique by changing some important parameters viz., rotations per minute, temperature of crystallization, while keeping the concentration of PETN constant as in solvent acetone. The synthesized mcPETN is characterized for its crystal structure and morphology. The enhanced performance to its macro sized PETN is reported herewith the results.

Keywords: mcPETN, Solvent anti-solvent crystallization, Crystal engineering, Impact sensitivity, Primary explosive.

INTRODUCTION

PETN is one of the high energy materials having oxidizer and fuel in the same molecule¹. PETN is used as a secondary explosive. Using a different primary explosive to initiate the secondary has its limitations such as, problems in synthesizing high sensitive primary explosives, heterogeneity etc. PETN is considered to have medium sensitivity towards the impact and electric shock.

Coarsening of PETN is the main drawback that limits PETN applicability in the detonators. Doping impurities has managed the problem of aging and coarsening². But the impurities may alter the thermal behavior of PETN^{3,4}. Specific surface area of PETN is the key parameter in choosing PETN in exploding bridge wire (EBW) detonator⁵.

High surface area PETN production through crash precipitation was reported by Pitchimani *et al.*,⁴. Changes in the material bulk alter the response towards impulses.

Single crystal of PETN was developed by Meiyu Zhai and Gregory B. Mc Kenna in 2016. The authors have explored the elastic property of PETN single crystal using nano-indentation test⁶. A brief study on application of PETN in demolishing works and under water blasts in the form of plastic explosives are dealt by Ahmed Elbeih⁷. Semtex is the combination explosive which is made by mixing PETN and RDX in different proportions⁸.

The crystal engineering techniques comes forth to play a key role in removing the problems that arise due to crystal defects. The crystal size

reduction is the technique we adopted in this work. The reduction in crystal size may enhance the surface area and change the morphology to less sensitive.

Solvent anti-solvent crystallization technique is one of the most adopted techniques because the crystals obtained are uniform in size and morphology. Achieving super saturated solution and finally the uniform crystals through the solvent anti solvent method is the basic principle⁹.

EXPERIMENTAL

Materials and Methods

The commercial macro PETN is obtained from the Premier Explosives Limited, Hyderabad, India. Acetone and distilled water are taken as solvent and anti solvent respectively in the ratio of 1:10. The commercial macro PETN is mixed with acetone to form a uniform solution. The above solution is added drop by drop to water. The fine

crystals of PETN are appeared in the water.

Characterization

Recrystallized final powder is characterized for average particle size (APS) and specific surface area (SSA) using Malvern instrument and Micromeritics instrument respectively. Crystal density of the final powder is calculated manually. Final powder is characterized further for its particle morphology through SEM. Olympus BX-51 instrument is used to get the SEM image.

Functional tests

The synthesized mcPETN is tested for its performance. Electric firing test is carried out at 5.2kV by placing 1g of mc PETN over an aluminium plate of 3mm thickness.

RESULTS AND DISCUSSIONS

This report included the recrystallization of mcPETN at two RPMs viz. 500, 1500 RPM, two

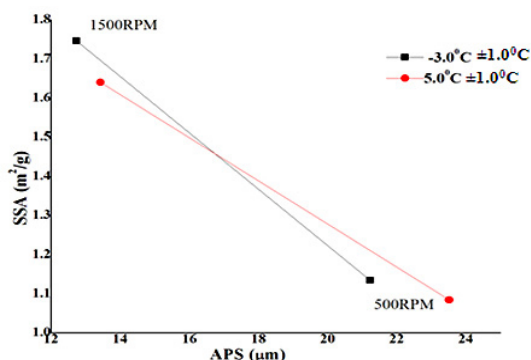


Fig. 1: APS vs SSA at different temperatures and different RPMs

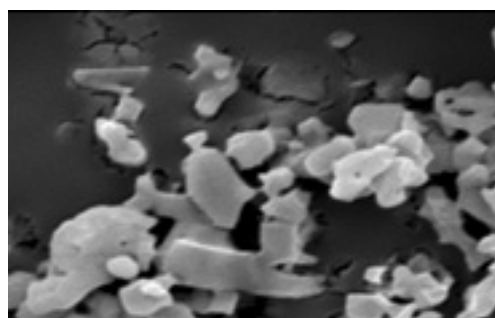


Fig. 2: SEM image of mcPETN

Table 1: Crystal densities, APS and SSA details of Macro PETN and mcPETN at different temperatures and RPMs

S.No	PETN (g)	RPM	Temperature (°C)	Crystal density (gm/cc)	APS (μm)	SSA (m²/g)
Macro PETN				1.76	446.4	0.052
01	1	500	-3.0 ± 1.00C	1.67	21.24	1.134
02	1	1500	-3.0 ± 1.00C	1.53	12.74	1.746
03	1	500	5.0 ± 1.00C	1.42	23.52	1.084
04	1	1500	5.0 ± 1.00C	1.69	13.43	1.640

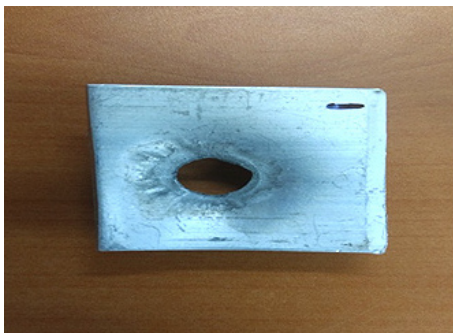


Fig. 3: Hole formed on aluminium plate in electric firing test.

temperatures viz. $-3.0 \pm 1.0^\circ\text{C}$ and $5.0 \pm 1.0^\circ\text{C}$. The crystal density, APS and SSA of commercial macro PETN and recrystallized mcPETN are tabulated in table 1.

The plot between APS and SSA (figure 1) shows the dependence relation between APS and SSA of mcETN.

The SEM image of mcPETN is given in figure 2. The agglomerations in the SEM image are possibly due to the presence of water. During the synthesis of mcPETN, the rods like crystals have changed their morphology to spherical, thus improving the SSA. The rods like crystals appeared in figure 2 are less in size when compared with the commercial macro PETN. The spherical crystals have high amount of hotspots in the solid matrix than the regular rod like crystals¹⁰.

The electric firing test results a hole on the aluminium plate with 3mm thickness as shown in figure 3, while the commercial macro PETN did not initiate the firing. Reduced APS and availability of extra SSA to the electric stimulus may have caused the explosion. The increased hotspots in the solid matrix of the mcPETN crystals are also be responsible for the reactivity towards low electric impulses (5.2kV).

CONCLUSION

The PETN crystallized during solvent antisolvent crystallization technique was observed to be in micro size. The APS, SSA graph has revealed the mcPETN observance. SEM image shows the conversion of rod like crystals to spherical crystals which may lead to the high SSA eventually to its activity towards low electric impulse. The replacement of metallic primary explosives with mcPETN in the manufacturing of armaments where the PETN is being used as a secondary explosive is proposed.

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