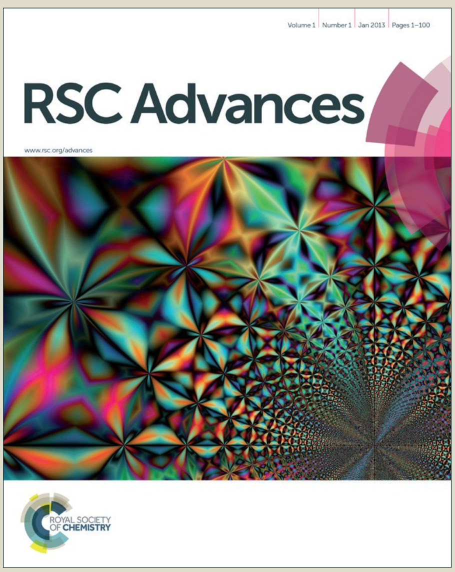
RSC Advances



Spherical 3-Nitro-1, 2, 4-triazol-5-one (NTO) based Melt-cast compositions: -Heralding a new era of Shock Insensitive Energetic Materials

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Abstract

3-Nitro-1, 2, 4-triazol-5-one (NTO) is an unique candidate among military high explosives and is explored as potential bomb fill with TNT in melt cast formulations. In the present study, we attempted to replace sensitive RDX with NTO having spherical morphology to develop a less hazardous, thermally stable shock insensitive composition. Bimodal mixture (150 and 25 μm in 70/30 ratio) of spherical NTO powders having superior flowability was chosen for formulations and achieved 60% of solid loading. Temperature sensitivity of the formulations was assessed by calculating the activation energy for the flow. Velocity of detonation and shock sensitivity of the composition were also determined. The study inferred that the spherical-NTO/TNT (60:40) was found to be 2.5 times shock and 2 times friction insensitive than Composition B which consist of RDX/TNT (60:40). Activation energy for the thermal decomposition was determined to assess the thermal hazards and vacuum stability test was carried out to ensure the storage life of composition.

Keywords: Activation energy, insensitive molecule, insensitive munitions, melt-cast compositions, thermally stable explosive, viscosity.

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1. Introduction

Energetic materials are inherently hazardous in nature and sensitive to numerous hazard stimuli which leads to accidents involving explosive filled munitions have come at a high cost in terms of weapons platforms, personnel and material. The intention of this research work on energetic materials is to develop safer, less hazard and high or moderate performance melt-cast formulations compared to Composition B which typically consist of 60% RDX and 40% TNT. The composition B is being universally employed over 60 years in munitions in several anti-armor warheads and shells as an explosive fill due to its high performance, ready availability and ease of processability. The high sensitivity to various stimuli and catastrophic explosions associated with this composition limits the realization of shock insensitive munitions.[1] Alternatively, in order to meet insensitive munitions (IM) requirements, shock sensitive RDX can be replaced in the existing formulations. The use of intrinsically insensitive candidates such as TATB, NTO, and FOX-7 etc., which are stabilized by extensive inter and intra molecular hydrogen bonding are the promising replacements for the RDX.[2]

3-Nitro-1, 2, 4-triazol-5-one (NTO) is an unique candidate among military high explosives for an effective insensitive munitions and a potential bomb fill in admixture with TNT under investigation.[3] Various NTO based melt-cast formulations such as Picatinny Arsenal Explosives (PAX), Ordnance System Explosives (OSX) and Insensitive Munitions Explosives (IMX)-101 (containing DNAN, NTO and NQ) and IMX-104 (containing 40% DNAN or TNT, 40% NTO and 20% RDX) etc., have been reported using TNT or DNAN as a binder.[4] Among these, IMX-104 composition involves the part replacement of RDX and the VOD determined to be 7190±200 m/s and 7410±100 m/s for DNAN and TNT respectively. Accordingly impact sensitivity of TNT based composition is higher than DNAN. Further, biodegradation and phytoremediation of IMX-101 formulations were also investigated by Richard et al.[5, 6] It is also inferred from the toxicological studies that NTO is non-toxic compared to RDX and TNT and Table S1 of supporting information lists the LD_{50} values of the studied explosives. Cliff and Smith et al developed RDX-free ARX-4002 melt-cast formulation consisting of NTO/TNT (50:50). Lower amount of solid explosive (NTO) in the reported formulation may be due to the usage of non-spherical NTO and also the solid loading can be significantly increased by the use of spherical NTO.[7] Large scale preparation of NTO involves crystallisation of NTO from water and this yields the irregular rods and jagged crystals.[8, 9] This irregular and undesired crystal morphology lead to high viscosity, poor processability and hence reduced solid loading. Our previous studies reported crystallisation process for the preparation of spherical-NTO (SNTO) of various particle sizes and characterisation of prepared powders. It was demonstrated that the use of spherical-NTO improves the mix fluidity of composite explosives significantly and hence the solid explosive loading. [10, 11] Further, it was also revealed that spherical crystals of explosives can improve insensitivity towards a sudden shock, performance, processability and packing density than non-spherical crystals.[12-14]

The present study is aimed to develop less hazardous shock insensitive melt-cast explosive formulations based on NTO by complete replacement of RDX (60%) in composition B. In order to realise higher solid loading, spherical NTO with good flow characteristics was identified and employed in the formulations. Anchor blade mixer was used for processing and the explosive charges were made using suitable moulds. Performance and hazard assessments like shock, friction and impact sensitivities were carried out using these charges. The study also reports the activation energy of thermal decomposition of SNTO/TNT and RDX/TNT (60:40) melt-cast formulations by Ozava and Kissinger methods to determine the thermal hazards of compositions. The thermo gravimetric study and vacuum stability tests have been carried out to asses the storage stability of compositions.

2. Experimental

2.1 Materials and Methods. All the reagents and chemicals used in the present study were of AR grade and used as such without any purification. Spherical NTO of specific particle size was prepared in-house by a cooling crystallization process from non-spherical NTO.[10] The bimodal mixture was made from 150 and 25μm powders in 70:30 weight ratio.

Calorimetric studies were undertaken on a Perkin Elmer DSC-7 instrument at four different heating rates 2, 5, 10 and 15°C/min under nitrogen atmosphere with 1mg of sample. Ozawa and Kissinger method was employed to calculate activation energy (Ea) of thermal decomposition reactions.[15-18] Thermo-gravimetric Analysis (TGA) was carried out on simultaneous thermal analyzer (TA instruments SDTQ600) at 5^{o}C/min under nitrogen atmosphere using open platinum cup. Morphological characterization was carried out in Optical Microscope (RAX-vision Y-coo series). The extent of sphericity is expressed by means of circularity which is determined by the following formula: (4πArea) / (Perimeter^2).

Particle size distribution was measured from Sympatech particle size analyzer in dry mode by applying Laser diffraction method and volume mean dia, D [4, 3] is reported in the present study. True density was determined by measuring the changes in pressure with helium gas displacement using gas pycnometer of Thermo Scientific Instruments. USP-II standard procedure was adopted to measure the tapped bulk density with the defined set of tapping procedure using Veego Industries, India make Tap density apparatus. Flowability properties like Carr's index (CI) and Hausner ratio (HR) were also calculated from the above measurements. All viscosity measurements were made using Brookfield viscometer (RV-DVII+ Pro) equipped with small sample adapter (SC4-27) where shear rate and stress can be measured. The sample was placed in a chamber and which was heated through a circulator. All measurements were made at 81, 83, 86 and 90^{o}C and at the shear rates varying from 17 to 59.5 s^{-1}.

In order to determine the chemical and thermal stability under extreme conditions and also to verify the compatibility among the explosives in the melt-cast compositions, the vacuum stability tester (Tirupati scientific industry, Calcutta) was used. A dried and weighed sample (5 g) of both RDX/TNT(60:40) and SNTO/TNT (60:40) was heated at 120°C for 48 hours and the volume of gases (mL/g) evolved was recorded and the experiments were repeated for their consistency.

2.2 Preparation of melt-cast formulations

The compositions were processed by the standard, melt-cast technique involving addition of bimodal spherical NTO to molten TNT under continuous stirring in a steam jacketed 7 liter anchor blade mixer at the temperature of 95ºC (Fig.1). The speed of the anchor blade and side propeller was kept at 50 and 70 rpm respectively. The mixture was stirred for about 20-25 min and then transferred to a suitable mould. The inner diameter of the mould in which casting of VOD charge was carried out is 35mm and length of the mould is 300mm. After cooling to ambient conditions, the charge was removed from the mould and machined to the required dimensions. The inner and outer diameter of mild steel shock sensitivity tube which contains main acceptor charge was 44 and 55 mm respectively.

2.3 Determination of Sensitivity Characteristics

Sensitivity and performance of melt-cast compositions have been determined by using standard methods. The fall-hammer method having 2kg drop weight by employing Bruceton staircase approach, impact sensitivity of the explosive compositions was determined. The results are expressed statistically in terms of 50% probability of explosion ($h_{50\%}$). Julius Peter's apparatus was used to determine the friction sensitivity and measurements were carried out in duplicate for confirmation.

Shock sensitivity was determined by the standard card gap test, using cellulose acetate sheet as an attenuator and CE pellet (Tetryl) as donor charge. The sheet thickness of cellulose acetate was varied until No-Go was observed on the witness plate while carrying out experiments with RDX and SNTO charges. The shock sensitivity of the melt cast explosive composition is expressed in terms of the minimum pressure of the shock wave which can initiate detonation. The critical pressure (P in kbar) developed across the cellulose acetate sheets which can detonate the explosive composition with 50% probability was determined from the following equation:

$$P\ (\text{kbar}) = 105e^{-(0.0358x)}$$

Where 'P' is the critical pressure in kbar and 'x' is the thickness of the cellulose acetate sheet as an attenuator in mm. The repeatability of shock sensitivity results were confirmed with consistent No-Go observations.

2.4 Determination of Velocity of Detonation (VOD)

Velocity of detonation is the performance parameter in melt-cast formulations and it was determined by employing pin ionization probe technique. Explosive charges were pre-inserted with pin type ionization probes which were twisted with enamel copper wire at predetermined points and made ready for detecting the arrival time of the detonation wave. An oscilloscope instrument (Make: YOKOGAWA DL9140, 1GHz) was used for data acquisition. Three charges of each formulation was subjected to the test and fired for the determination of the velocity of detonation. VOD is reported after three firings of each composition and expressed as an average of three trials.

3 Results and Discussion

3.1 General characterization and selection of powders for compositions

The particle size and its distribution are very essential and play an important role in the field of high explosives and rocket propellant formulations. The size distribution of particles is well understood with the help of span. There are several measures of absolute width one can derive from given cumulative distribution. One common measure is the span, D_{90} - D_{10}. A dimensionless measure of width is the relative span defined as span/D_{50}. The narrower a distribution is the more closely the absolute measures of width approach zero. Optical microscopic images of spherical-NTO having 150 and 25µm particle size obtained from the controlled cooling crystallization with water and NMP as solvent system are shown in Fig.2. Table 1 presents the particle size and span data. It is 0.63 and 0.69 for spherical-NTO of 150 and 25µm respectively which indicates narrow particle size distribution. Fig.3

shows the morphology of RDX (185μm) and the span (0.73) observed to be relatively broad distribution. Similar to the particle size, shape is also fundamental property of the material in powder technology which affects many processing parameters in final formulations. In order to measure the shape of particle, circularity is a measure of sphericity of particles. Circular objects will have circularity of 1 while other shapes will have less than 1 and deviation from one gives the degree of sphericity. Spherical-NTO having D (4, 3) 25 and 150μm found to have the circularity between 0.91 to 0.86 which indicates that the particles are very close to spherical shape.

It is important to control the size distributions of particles in attaining high density and also to achieve maximum packing density in the formulations which further increases the amount of solids per unit volume.[19, 20] Further, the size along with shape of the particle plays a key role in obtaining the flowability of powders. Our earlier studies on spherical NTO powders demonstrate the flowability parameters such as Carr's Index (Compressibility Index) and Hausner ratio (HR) which are the simple measures to describe the complete flow properties of material.[21-24] To attain the specific density many combinations of distribution are preferable. In order to achieve efficient packing the various ratio of coarse (150μm) to fine (25μm) powders were screened and based on the high density and flowability, 70:30 ratio (Coarse to fine) was chosen and analysed for their particle size and further used for formulation studies (Fig.4). The optimized bimodal mixture obtained maximum tapped bulk density i.e.1.09g/cc with Carr's index less than 15 and Hausner ratio less than 2 (Fig.5). True density is a fundamental parameter contributing to the characterization of a product and directly proportional to the performance of an explosive and also helps to identify different polymorphs of particular molecule.[25] True density of virgin explosives were determined and presented in Fig.5. The combination of bimodal mixture of SNTO (70:30) resulted to give 1.892 g/cc which is in between of SNTO 150μm (100%) and SNTO 25μm (100%) which indicates that efficient packing has been occurred.

3.2 Physical and flow characteristics of the composition

In case of melt cast formulations pourability is the deciding processability criteria and our earlier studies confirmed the superior flowability of NTO/TNT over bench mark RDX/TNT composition.[26] The physical appearance of prepared melt-cast compositions of SNTO/TNT and RDX/TNT (60:40) was found to be smooth. The void free solid blocks were further witnessed by true density measurements as presented in Table 2 and these compositions have been used for further detail characterization. True density of the SNTO/TNT (60:40) is higher than the Composition B.

Rheological behaviour of a material is greatly affected by the temperature and precise control of temperature is a major importance in viscosity measurements. It is also vital for their safety during handling and production. Our previous study reported that the temperature dependent flow phenomenon of melt-cast formulations and described by Arrhenius equation of ideally viscous materials.[26] It is observed that, SNTO/TNT (60:40) composition exhibited temperature independent behaviour (Fig.6) (No significant variation in viscosity). Viscous flow phenomenon involves a thermally activated rate process and in order to move molecules to an adjacent vacant site they must overcome an energy barrier. By applying Arrhenius relationship, activation energy for flow has been obtained. Fig.7 compares the activation energy for the flow of the compositions. RDX/TNT based composition requires about three times higher activation energy for flow than SNTO/TNT (60/40). This clearly indicates that the role of chemical composition and nature of material on the activation energy for flow and also indicates the relative temperature susceptibility of the different compositions. From this study, it can be inferred that the SNTO/TNT composition is found to be insensitive to temperature and hence, processing can be done nearly at the melting temperature of TNT. In contrast to SNTO, the dependency is high in the case of RDX based bench-mark composition, which demands higher processing temperature.

Sedimentation of solid in any liquid matrix plays a crucial role especially in melt-cast compositions. Sedimentation rate of solid explosive was studied for both compositions at 86^{o}C under shear rate of 59.5 s^{-1} for a period of 1hour. Viscosity was noted at an interval of 5min and plot of time versus viscosity is shown in Fig.8. The study reveals that the rate of increase of viscosity is high for RDX based composition and it may be due to the non-uniform distribution of RDX. Increase of viscosity is low in case of SNTO/TNT (60:40) and it is mainly due to the stronger interaction of NTO and molten TNT which kept the dispersion more stable and hence lower sedimentation rate.

3.3 Hazard Assessment and Vacuum Stability Studies

3.3.1 Thermal assessment of SNTO/TNT and RDX/TNT (60:40) compositions

The importance of understanding the thermal behaviour of explosives is prime in the explosive field for handling and safety during manufacturing of compositions. In order to review the thermal hazard of the final compositions, decomposition study was carried out in differential scanning calorimeter and the maximum of the decomposition temperature (T_{max}) of both compositions at the heating rate of 10^{o}C/min is reported in Table 3. It is clearly understood from the data that spherical NTO based composition thermally more stable than Composition B. The activation energy for melt-cast compositions, RDX/TNT (60:40) and SNTO/TNT (60:40) has been determined by using the generated data at different heating rate 2, 5, 10 and 15°C/min (Fig. S1 & S2). The profile of the thermal curves of RDX/TNT (60:40) and SNTO/TNT (60:40) composition was similar. Notably, in all thermal experiments, both samples exhibited melting of TNT initially at about 78 ºC, while an exothermic peak was observed from 254ºC to 272 ºC for NTO/TNT and 227 to 249ºC for RDX/TNT. The DSC data showed that the maximum of the decomposition temperature (T_{max}) of the material was increased with the increase in heating rate.

The activation energy for decomposition of compositions was computed by Ozawa and Kissinger method.[15-18] Arrhenius plots of these compositions are shown in Fig.9a, 9b & Fig.10a, 10b and calculated data are given in Table 4 & 5. Activation energies of these compositions are 185 and 259kJ/mol for RDX and SNTO based compositions respectively. These values are not significantly varied while calculating from the above methods. Higher activation energy of SNTO/TNT composition indicates high thermal stability and relatively safe at elevated temperatures compare to composition B. This increased thermal stability may be attributed to the existence of hydrogen bonding stabilised layered structure of NTO.

The thermo gravimetric analysis (TGA) of the melt-cast compositions is given in Fig.11. RDX/TNT (60:40) composition starts to show weight loss within a temperature range of 94.8 to 241.7°C in two steps. The loss in weight for the composition in the first step is found to be 28.8% in the temperature range of 94.8 to 178.4°C which corresponds to loss of TNT, while 58.9% loss in weight was observed in the second step corresponds to RDX in the temperature range of 178.4 to 241.7°C. SNTO/TNT (60:40) composition was also decomposed in two steps as shown in Fig.11. It starts to show weight loss within a temperature range of 104.5 to 264.2°C. The first step shows 38.9% weight loss in the temperature range of 104.5 to 185.6°C which corresponds to TNT, whereas 63% loss in weight was observed from temperature range of 185.6 to 264.2°C corresponds to NTO. This study clearly examines that the weight mixtures of SNTO/TNT and RDX/TNT from temperature range 104.5 to 264.2°C and 94.8 to 241.7°C indicated that each species enhanced the decomposition of the other. This shows that the NTO containing formulations are found to be thermally stable as brought out by the thermal decomposition studies and this composition is less susceptible to storage temperature compare to RDX/TNT compositions. Both calorimetric and weight loss studies brought out that SNTO/TNT (60:40) composition shows good thermal and storage stability.

3.3.2 Vacuum stability assessment of melt-cast compositions

Vacuum stability test was carried out primarily to find out the compatibility of ingredients used in the compositions. The test was performed for both RDX/TNT (60/40) and SNTO/TNT (60/40) compositions at 120°C for 48 hours. The volume of gases evolved from the composition was determined to be 1.56 and 1.08 ml per five gram of test material for RDX/TNT and SNTO/TNT compositions respectively. This is well within the acceptable limits that clearly indicated spherical-NTO and RDX compositions are well compatible with TNT. Hence it is expected to possess good storage stability.

Thus the SNTO/TNT (60:40) melt-cast compositions exhibit better thermal and storage stability even under extreme conditions. The vacuum stability results were also corroborated with the TGA and DSC analysis.

3.3.3 Sensitivity Assessment of melt-cast compositions

To realise less hazardous and insensitive munitions (IM's), shock sensitivity is an important criterion in melt-cast formulations. The typical set-up used for the determination of shock sensitivity is shown in Fig.12. 10mm thickness mild steel sheet was used as a witness plate on which acceptor charge consisting of SNTO/TNT was placed. 136g of booster donor charge separated by attenuator sheet which consisting of cellulose acetate was assembled over the acceptor charge. Shock sensitivity of SNTO/TNT composition is 51.3kbar which is significantly higher than the corresponding RDX/TNT based composition (Table 3). In explosive formulations, friction insensitivity plays an important role and spherical NTO based compositions are almost two times friction insensitive than that of bench-mark composition. However, SNTO/TNT composition was found to be slightly sensitive to impact stimuli compared to composition B.

Overall, melt-cast compositions with spherical-NTO possessing 60% solid loading was developed and realised composition exhibiting 2.5 times more shock insensitive and 2 times friction insensitive compared to RDX based Composition B. Insensitivity of spherical NTO based compositions may be attributed to the layered crystal structure of NTO unlike RDX.

3.4 Evaluation of explosive properties of SNTO/TNT and RDX/TNT compositions

The explosives characteristics were also determined for these formulations and compared with the standard explosive TNT are given in Table 3. Determined the velocity of detonation by ionisation probe method is significantly lower than the benchmark composition B. This may be due to the poor charge density achieved in the case of NTO composition compared to its estimated theoretical maximum density. Since the morphology of NTO is spherical, it may be possible to increase the solid loading up to 75% with efficient vacuum mixing and casting. Hence, we believe that the NTO composition with increased solid loading will have enhanced performance combined with superior insensitivity. This study further recommends using the combination of RDX (or Reduced shock sensitive RDX) and NTO to achieve better performance along with the shock insensitivity.

4. Conclusions

Thermally less hazardous and shock insensitive melt-cast composition is developed by replacing sensitive RDX with spherical NTO. Because of the spherical morphology, NTO loading is proved to 60% by simple processing methodology. Activation energy for the thermal decomposition of NTO/TNT is determined to be high compared to RDX/TNT and hence relatively safer than existing RDX/TNT composition. Superior processability also realised with NTO based composition. Further, the weight loss and vacuum stability studies also indicated that SNTO/TNT composition exhibited less susceptibility to storage temperature and possess good storage life. SNTO/TNT composition is found to be shock insensitive (51.3 kbar) and realised 2.5 times shock insensitive compare to RDX/TNT (18.7 kbar) composition B. The composition is also found to be friction insensitive. Though the velocity of detonation of SNTO/TNT is relatively lower than benchmark, but it can be realised by increasing NTO content or by part replacement of reduced sensitivity RDX. Overall, this study on NTO/TNT based melt-cast composition concludes the realisation of less hazardous and thermally stable shock insensitive composition for the future insensitive munitions.

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Tables

Table 1

Particle size and its distribution of RDX and NTO

Product	D (4,3)	Span*
RDX	185µm	0.73
Spherical NTO	150µm	0.63
Spherical NTO	25µm	0.69

*Span = $(D_{90}-D_{10})/(2\ D_{50})$

Table 2
True density of virgin explosives as well as melt-cast compositions

Composition	True Density (g/cc)
RDX (100%)	1.798
TNT (100%)	1.616
RDX/TNT (60:40)	1.717
SNTO/TNT (60:40)	1.741

Table 3
Performance and sensitivity data of melt-cast compositions

Composition	Theo. Max Density (g/cm^3)	Experimental density (g/cm^3)	VOD (m/s)	Sensitivity to Various Stimuli			
				T_{max} (^{O}C)	Shock (kbar)	Friction (kg)	Impact h_{50} (cm)
SNTO/TNT (60/40)	1.79	1.65	7100	266	51.3	36	72
RDX/TNT (60/40)	1.74	1.68	7900	241	18.7	14.8	99

Table 4
Activation energy of decomposition of RDX/TNT (60:40) by Ozava & Kissinger Method

Heating rate (β) (^{o}C/min)	Tm (K)	T^2m	1/Tm (K)	log β	ln β	ln (β/T^2m)
2	500.52	2.50×10^{5}	1.99×10^{-3}	0.3010	0.6932	-11.7381
5	505.62	2.55×10^{5}	1.97×10^{-3}	0.6990	1.6094	-10.8421
10	514.17	2.64×10^{5}	1.94×10^{-3}	1	2.3026	-10.1825
15	522.31	2.72×10^{5}	1.91×10^{-3}	1.1761	2.7081	-9.8085

Table 5
Activation energy of decomposition of SNTO/TNT (60:40) by Ozava & Kissinger method

Heating rate β (^{o}C/min)	T (^{o}C)	Tm (K)	T^2m	1/ Tm (K)	log β	ln β	ln (β/T^2m)
2	254.02	527.02	2.77×10^{5}	1.89×10^{-3}	0.3010	0.6932	-11.8413
5	261.93	534.93	2.86×10^{5}	1.86×10^{-3}	0.6990	1.6095	-10.9548
10	266.47	539.47	2.91×10^{5}	1.85×10^{-3}	1	2.3026	-10.2786
15	272.21	545.21	2.97×10^{5}	1.83×10^{-3}	1.1761	2.7081	-9.8943

Figures

Fig.1. Steam Jacketed Anchor Blade Mixer

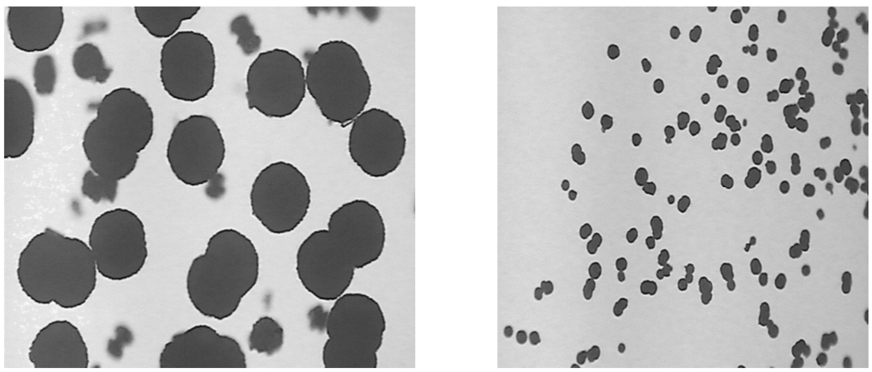

Fig.2. Optical microscopic images of Spherical-NTO 150 and 25μm

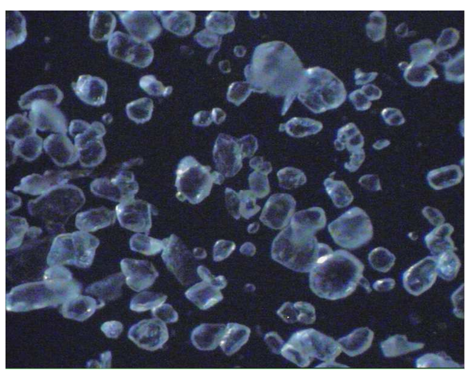

Fig.3. Optical microscopic image of RDX

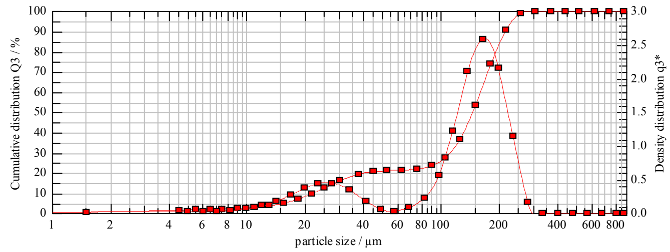

Fig.4. Particle size analysis of Bimodal Mixture of SNTO (70:30)

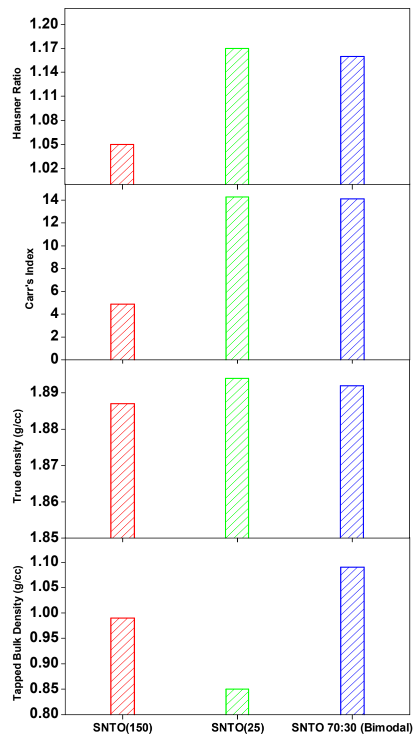

Fig.5. Flowability parameters of virgin SNTO and bimodal mixture (70:30)

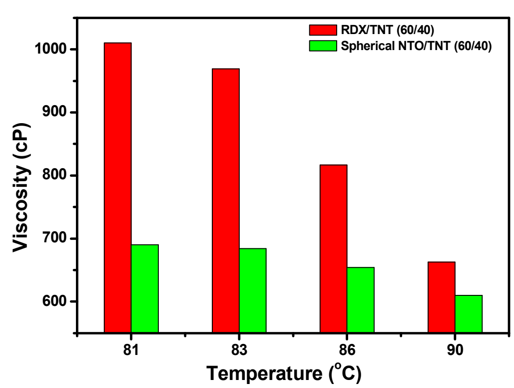

Fig.6. Temperature dependant flow behaviour of Melt-cast compositions at shear rate of $59.5 s^{-1}$

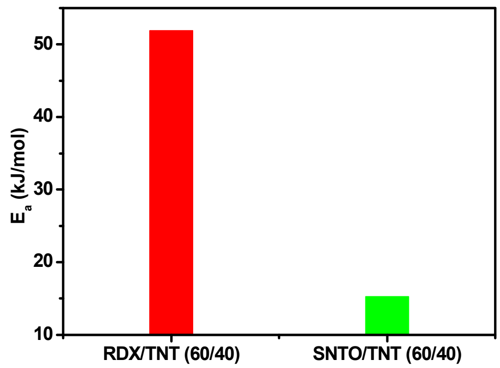

Fig.7. Activation energy for flow of melt-cast compositions

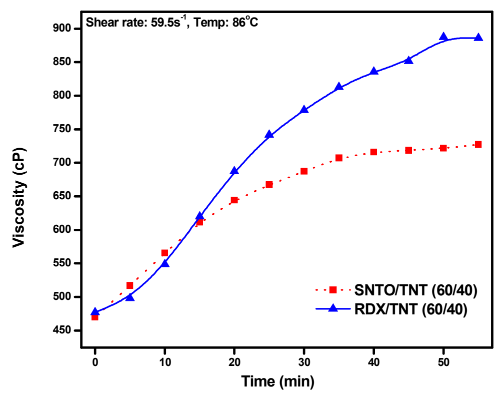

Fig.8. Plot of time versus viscosity of RDX/TNT and SNTO/TNT compositions

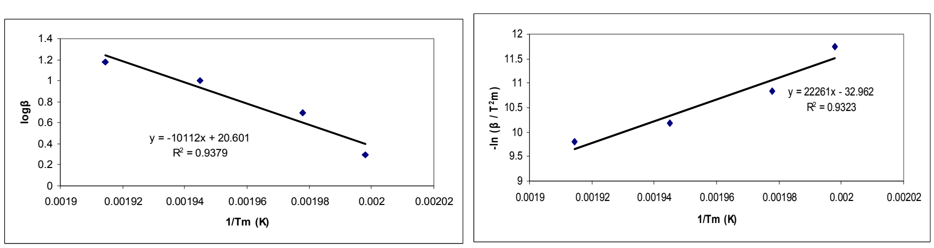

Fig.9a & 9b. Activation Energy of decomposition (RDX/TNT 60:40) by Ozava and Kissinger Method

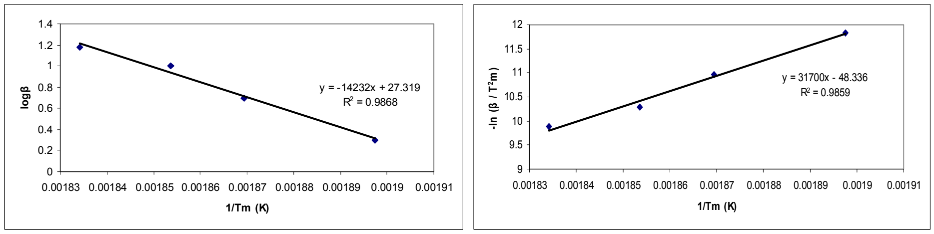

Fig.10a & 10b. Activation Energy of decomposition (SNTO/TNT 60:40) by Ozava and Kissinger Method

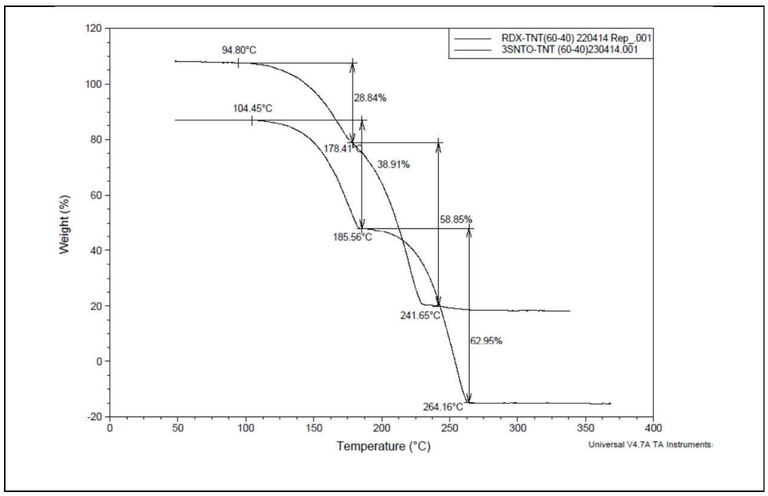

Fig.11. TGA profile of RDX/TNT and SNTO/TNT (60:40)

Fig.12. Trial set up for Shock Sensitivity Test