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Research paper

Influence of Temperature on the Thermal Expansion and Other Physical Properties of Pressed RDX–Wax Explosive Pellets

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Abstract: Thermal expansion is an intrinsic property of every material. The thermal expansion of LX-17, PBX-9502 and ultrafine triaminotrinitrobenzene (TATB) had already been reported; however, there are few reports on the expansion of RDX-based compressed explosives. In the present work, the thermal expansion of RDX associated with a polymeric environment of paraffin wax, in the ratio of 95% and 5%, was investigated using a thermomechanical analyzer (TMA). Fourier Transform IR (FTIR), Differential Scanning Calorimetry (DSC) and a Thermal Property Analyzer (TPA) were used to characterize and study the thermal properties of pressed RDX/wax explosive beads. The values of the coefficient of thermal expansion (CTE) and linear thermal expansion were measured and analyzed in the temperature range from –100 to 100 °C. Volume and density changes over the entire temperature range were evaluated. The irreversible growth of RDX-based compressed explosives was also observed. The results are related to the growth of RDX/wax-based explosive pellets due to repeated thermal stress.

Keywords: compressed explosive, thermal expansion, density change, coefficient of thermal expansion, CTE

1 Introduction

1,3,5-Trimethylenetrinitramine (RDX) was first synthesized in 1899. Since then, its use has been limitless in the form of civilian and various military applications. The production of high-energy materials by compression molding is preferred over the melt casting method. Compressible high-energy materials, such as RDX/wax in the form of pellets developed by compacting RDX granules in a polymeric environment [1], are widely reported in the literature. The material produced fulfills the requirements for mechanical and thermal stability. The thermal stability of RDX in different compositions has been extensively studied and described in the literature [2].

The thermal expansion of solids is a material property that explains the increase in size in relation to an increase in temperature of a material. Thermal expansion is well described in the literature [3] and is due to an increase in the average separation of atoms/molecules caused by the increased kinetic energy due to the increased temperature of the material. The measurement of thermal expansion is very important in the field of explosives. High-energy materials used by the military are based in different geographical locations and these explosives are exposed to different temperature ranges, resulting in the expansion/contraction of the explosives. The energetic component of high-energy materials is commonly coated with an inert binder to make the composition compressible and to reduce the sensitivity of the energetic component.

One of the most used explosives is the compressed explosive [4] and there are many compressible explosive compositions, such as HMX/wax, HMX/polyurethane, RDX/polyurethane, RDX/wax, RDX/Viton, *etc.* Compressed explosive pellets are used in warheads both as the main charge and as a booster. The details for the production of RDX/wax explosives were not publicly available. The US government's National Defence Research Committee published document [1] on the production of wax-coated RDX compositions in 1946. Thermal studies of explosive compositions such as TATB-based compressed explosives, boron, PBX 9502, TKX-50, propellants, *etc.* [7, 9-14] have been well documented by researchers. Earlier efforts were devoted to the study of irreversible rocket growth in TATB-based compositions and CTE (coefficient of thermal expansion) studies on HMX, FOX-7, TATB [5], PBX 9502 [7] and other explosive compositions. Studies on the thermal decomposition of RDX using various techniques [2] have been reported; however, a detailed study on the thermal behaviour of RDX/wax (95/5) over a wider temperature range is not available.

The thermal parameters of various explosives, such as HMX, FOX-7, PBX-9502, PBX-9503 *etc.* [2, 5], were analyzed for parameters such as thermal decomposition, glass transition temperature, CTE, phase transitions, linear expansion, by Differential Scanning Calorimetry (DSC), Thermogravimetry (TG), Differential Thermal Analysis (DTA), Fourier Transform Infrared Spectroscopy (FTIR), Thermomechanical Analysis (TMA), *etc*. RDX/wax compositions in the ratios 95/5, 92.5/7.5, 90/10, 88/12 have been optimized for different military applications. RDX/wax in the ratio 95/5 is one of the most optimized compositions for military applications. The thermal analysis of RDX/wax in the various compositions is only available for a limited temperature range, and there is insufficient information on the thermal expansion of RDX/wax (95/5) in the lower and upper temperature ranges.

The thermal parameters of explosive pellets can be evaluated, and their applicability can be correlated. In the present work, the thermal properties and their associated effects on the linear expansion, volume and density changes of RDX/wax (95/5) explosive pellets were investigated in a wider temperature range using TMA, DSC, FTIR and TPA (Thermal Property Analyzer).

2 Preparation and Evaluation of Explosive Charges for Detonation Velocity

2.1 Raw materials

The explosive pellets were prepared using RDX grade–I from Munition India Limited (MIL) with an average particle size of 100 µm and melting point of 205 °C, and was used as the main ingredient of the composition. Grade–I Special No. 9 kerosene-wax was used as the binder in the composition. The composition of RDX/wax in the ratio of 95% and 5% was used for the pellets.

2.2 Production of explosive pellets

To produce explosive charges, the composition should be compressible. The coating of the energetic particles of RDX with wax serves as a binder for compactness, but also makes the explosive insensitive, *i.e.* safer. The RDX/wax compositions were produced with a coating of wax with a nominal wax content of 5%. The wax-coated RDX was produced by adding an emulsion of wax in water and oleic acid to a slurry of RDX in the presence of dilute sulphuric acid (H_2SO_4). The filtered, wax-coated RDX was dried to ensure complete removal of the water content in the composition. The wax-coated RDX is a pressable composition for producing explosive pellets [1].

The explosive pellets are produced by pressing the RDX/wax composition in a mould, with an inner diameter of 0.84 cm using a plunger with an outer diameter of 0.83 cm. A pressing force of 350 kg/cm^2 was exerted by the plunger and the load was maintained for 8-10 s. Separation of the RDX crystals from each other, the presence of air cavities and the polymer layer contribute to the compactness of the explosive material. The compactness of the RDX/wax also depends on the applied load, the type of binder, the dwell time, etc. Three sets of pellets weighing 1155, 1100 and 1033 mg, 5 pellets each, were pressed in the same mould under ambient conditions. A limiter is placed between the flask head and the top of the mould to ensure the fixed sample length.

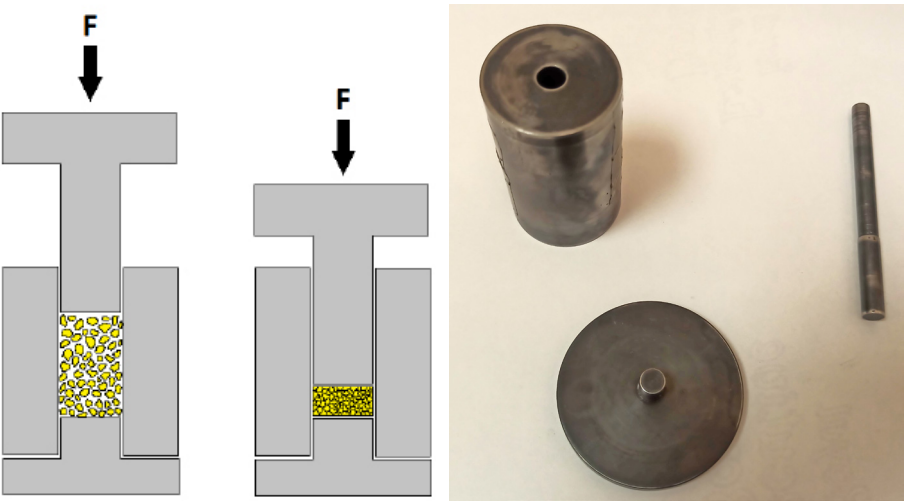


Figure 1. Experimental setup for the pressing of RDX/wax in the mould

Each pellet sample had a diameter of 0.845 cm and measured lengths as 1.311, 1.30 and 1.29 cm, and were categorized as sample 1, sample 2 and sample 3. The volume of the prepared specimens was 0.735, 0729 and 0,723 cm^3, respectively for sample 1, sample 2 and sample 3, resulting in three different densities of the pellets. Three pellets were made for each density. The densities obtained in the three sets of pellets were 1.57, 1.50 and 1.43 g/cm^3. The details of the samples are listed in Table 1.

Table 1. Characterization of the samples

Sample	Length [cm]	Diameter [cm]	Mass [g]	Volume [cm^3]	Density [g/cm^3]
1	1.311	0.845	1.1547	0.735	1.57
2	1.30	0.845	1.100	0.729	1.50
3	1.29	0.845	1.030	0.723	1.43

3 Characterization of Explosive Pellets

3.1 FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) is a technique for analysing the infrared absorption or emission of a material. Here, the transmission spectra of pure RDX and the RDX/wax sample were used for analysis (Figure 2). The FTIR spectrum of wax coated RDX was recorded in the frequency range of 4000-450 cm^{-1}. The main characteristic peaks observed correspond to the peaks described in the literature. The main bands can be assigned to NO_2 (1588 cm^{-1}), NO_2 and N–N (1262 cm^{-1}), ring stretching band (1037 cm^{-1}), δ NO_2 and NO_2 (943, 779 cm^{-1}), N–NO_2 axial and equatorial stretch (1588 cm^{-1}), C–N stretching (1421 cm^{-1}) and NO_2 bending (1350 cm^{-1}). FTIR analysis revealed that no impurities were added during the preparation of the high-energy material. On comparing the RDX/wax FTIR spectrum with that of pure RDX the characteristic peak at 3073 cm^{-1} (C–H) remained unchanged indicating the presence of RDX. The presence of wax is clearly seen at 2916 cm^{-1} (aliphatic C–H), which is absent in the FTIR spectrum of pure RDX .

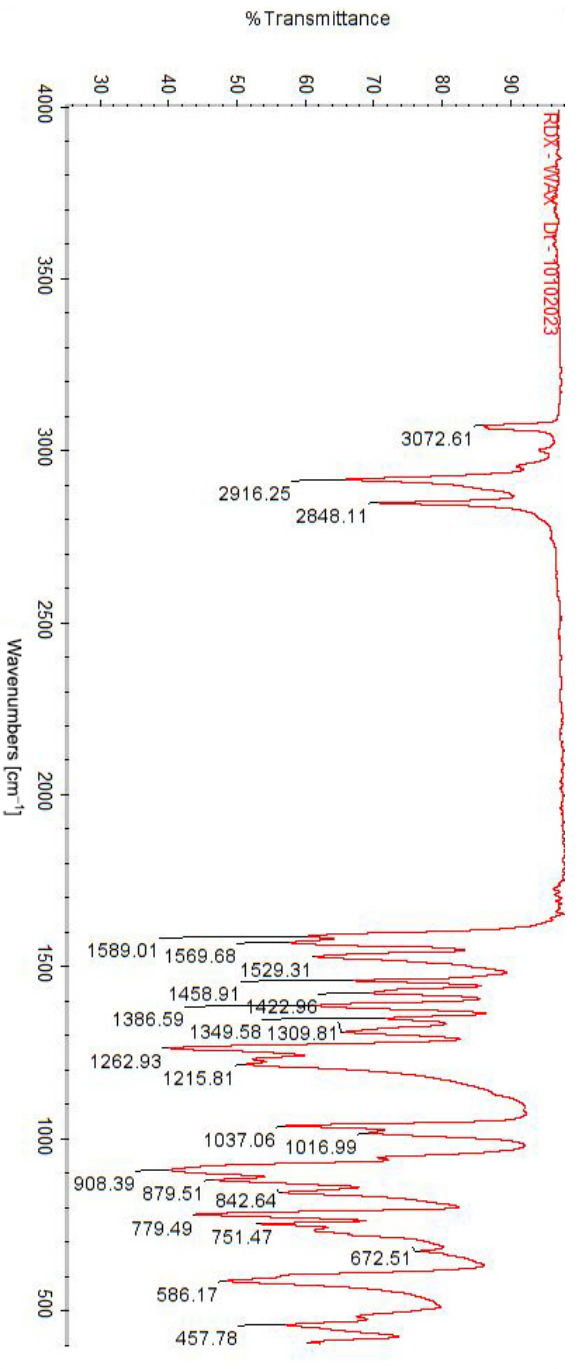
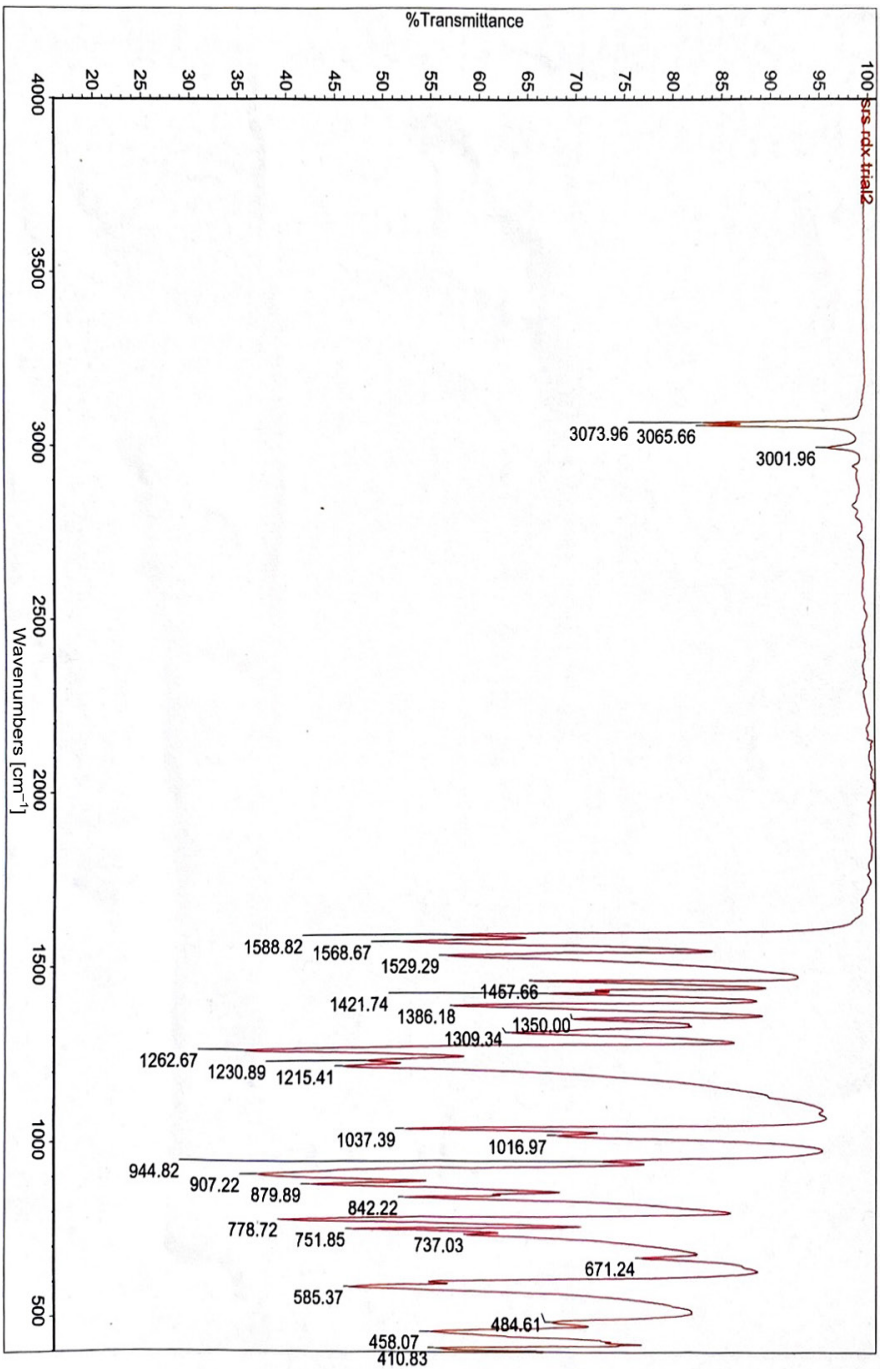


Figure 2. FTIR spectra of RDX (a) and RDX/wax (95/5) (b)



(a)

3.2 Thermal Property Analysis

The Thermal Property Analyzer (TPA) model FL3000 from Anter Corporation, USA, was used to measure the diffusivity, specific heat and thermal conductivity of the RDX/wax sample. Three RDX/wax sample pellets with diameter 1.2 cm and thickness 0.15 cm were produced. These pellets were sprayed with graphite to simulate a black body, as the coating improves the absorption and emission properties and serves to improve the signal-to-noise ratio, resulting in more accurate measurement data. The measurements were carried out at 32 °C. The results are shown in Table 2.

Table 2. Diffusivity, specific heat and conductivity of RDX/wax (95/5)

Temperature [°C]	Diffusivity [$cm^2 \cdot s^{-1}$]	Specific heat [$J \cdot kg^{-1} \cdot K^{-1}$]	Conductivity [$W \cdot m^{-1} \cdot K^{-1}$]
32	0.0017	815.5	0.23

3.3 DSC analysis

DSC was performed on a TA Instruments, model Discovery 25 instrument, in the temperature range of 50-400 °C. The sample quantity of 0.5 g was placed in an hermitically sealed aluminum pan in a nitrogen environment. The nitrogen environment was maintained by purging nitrogen at a flow rate of 50 mL/min. A heating rate of 10 °C/min was maintained throughout the complete heating range. The DSC curves of RDX/wax (95/5) obtained showed a sharp downward peak indicating an endotherm due to absorption of heat resulting from the melting of RDX. The DSC curves for RDX/wax (95/5) exhibited the endothermal onset temperature as 204 °C, indicating the transformation of RDX into the liquid phase with a peak temperature of 205 °C. The RDX started to decompose at an onset temperature of 217 °C and with an exothermic peak at 239 °C. This indicated the thermal stability of the wax-based binder on RDX. The results of thermal decomposition and thermal stability are like those claimed by other researchers [6]

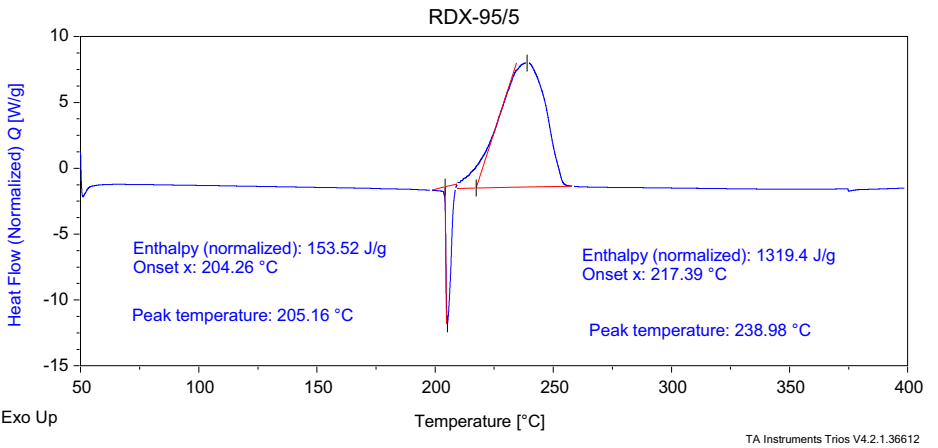


Figure 3. DSC of RDX/wax (95/5)

3.4 Thermal mechanical analysis

The thermal cycling was performed on a LINSEIS Thermal Mechanical Analyzer (TMA), model PT1000, with a measurement sensitivity of 2 nm. The sample, length 1.3 cm and diameter 0.845 cm, was placed on a quartz sample holder inside a quartz tube. The sample was conditioned in the complete temperature range of –100 to +100 °C, and measurements were taken. A constant thermal rate of 5 °C/min was maintained during cooling by liquid nitrogen and the same during heating in the furnace. A dwell time of 30 min was maintained at the extreme maximum and minimum temperatures and at room temperature for absorption of heat. The sample was under a load of 100 mN during the entire experiment. After reaching +100 °C, the sample was allowed to cool naturally to 40 °C, and afterwards the next cycle was initiated. Each sample was subjected to three thermal loadings from –100 to +100°C. The resulting values of TMA were the basis for the calculation of linear thermal expansion, volume expansion, and density variation over the entire temperature range

4 Results and Discussion

4.1 Coefficient of thermal expansion (CTE)

The coefficient of thermal expansion is a material property that indicates the expansion or contraction of a material with a change in temperature. The CTE is defined [15] as the change in length per unit length for a unit change in temperature.

$$\alpha_l = \frac{\Delta L}{L_0 \Delta T} \tag{1}$$

The CTE study of HMX, PBX 9502 and other TATB-based explosives [5, 16-18] is well documented by many researchers. These researchers measured the CTE values of these explosives in the elevated temperature range up to 250 °C and down to –60 °C in the lower temperature range. Thermal studies of pressed RDX/wax (92.5/7.5, 86/14) [4, 19] have been reported; however, information on the 95/5 composition is not well documented in the public domain. The CTE value of pressed RDX/wax (95/5) explosive pellets were recorded for each sample with a unique density and each sample consisted of at least 3 specimens. The average CTE value of all three samples was calculated for each density. The coefficient of thermal expansion of all three samples with different densities of pressed RDX/wax pellets was plotted against temperature. The CTE studies were performed in the temperature range from –100 to +100 °C and values were recorded and plotted over the entire temperature range.

The graph is approximately linear over the entire temperature range, except for the range at 20 °C. This is the reference temperature for measuring the change in length. The graph indicated that the CTE values are dependent on the density of the sample under observation. The value of the coefficient increases with temperature. The decrease in the graph is due to the softening of the wax near 50-60 °C, as the paraffin wax has a melting point of 44-68 °C. The pattern of the curves remains the same for all samples.

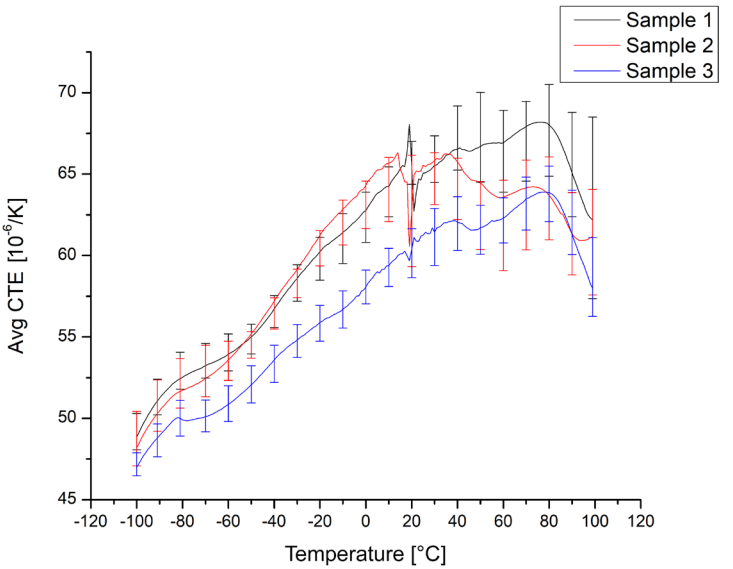


Figure 4. Variation of CTE of explosive pellets with temperature

4.2 Linear expansion with temperature

Thermal expansion with temperature is a phenomenon due to the increase in the kinetic energy of atoms/molecules [3, 10] of materials, which leads to more and more vibration; hence a greater average separation of atoms, resulting in a difference in the length of the sample studied. Thermal expansion of many explosive compositions, such as LX-17, PBX-9502, TATB-based explosives, pressed RDX/wax in the compositions (92.5/7.5, 86/14) has been presented by researchers [13, 14, 18-20]. In the present work, the linear thermal expansion of RDX/wax (95/5) was investigated in the temperature range from –100 to +100 °C.

The measurement of the change in length of the material is based on Equation 2.

$$\frac{\Delta L}{L_0} = \alpha_l \Delta T \tag{2}$$

where α is the coefficient of linear expansion. In the lower temperature range from –100 to +20 °C, the change in length, *i.e.* the shrinkage of the material, is higher at lower densities than at higher densities. This is due to the presence of air gaps in the pellets, and indicates the porous nature of the material. Also, the higher density material expands more than the lower density material in the

temperature range of 20 to +100 °C. This information is very important as the explosive pellets are used in warheads which are exposed to temperature stress when used in different geographical locations. As the explosive pellets are housed in metal fittings, the expansion or contraction of the explosive pellets can cause the warheads to stick to or become detached from the fittings.

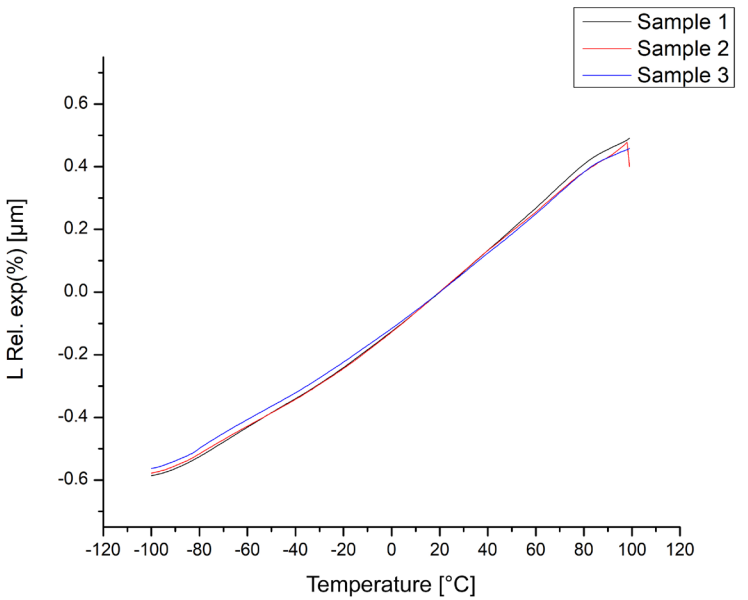


Figure 5. Variation of linear expansion of the explosive pellet with temperature

4.3 Volume of a sample

The data from TMA was used to measure the volume of the sample over the temperature range from –100°C to +100 °C. The volume of three samples was calculated using Equation 3:

$$\Delta v = \beta V \Delta T \tag{3}$$

in the case of an isotropic material. The pressed RDX/wax (95/5) is considered an isotropic material because there is no specific orientation of the RDX crystals during pressing.

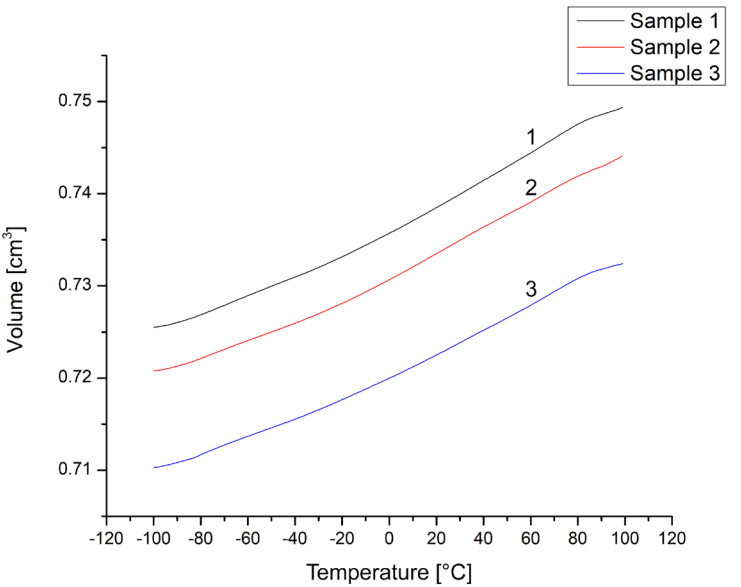


Figure 6. Variation of volume of pellet with temperature

The volume of the samples was calculated at each temperature in the range from –100 to +100°C. The volume was plotted against temperature for all three samples with different densities, and Figure 6 shows that the slope is different in the lower temperature range from that in the upper temperature range of the volume plot. At the macro level, however, the overall curve appears to be linear. The representation of the volume change of the material in relation to the volume of the material at 20 °C is shown in Figure 7. The overall volume change plot can be analysed in two different zones, namely –100 to +20°C and 20 to +100 °C. It can be seen from Figure 7 that the volume change in the range from –100 to +20 °C is different from that in the temperature range from 20 to +100 °C. The volume change is higher in the lower temperature range than in the upper temperature range. This is consistent with the above-mentioned linear thermal expansion in lower and higher temperature ranges. The contraction rate of the material in the lower temperature range is higher than the expansion of the material in the upper temperature range. This is due to the porous nature of the material and the presence of wax in the explosive.

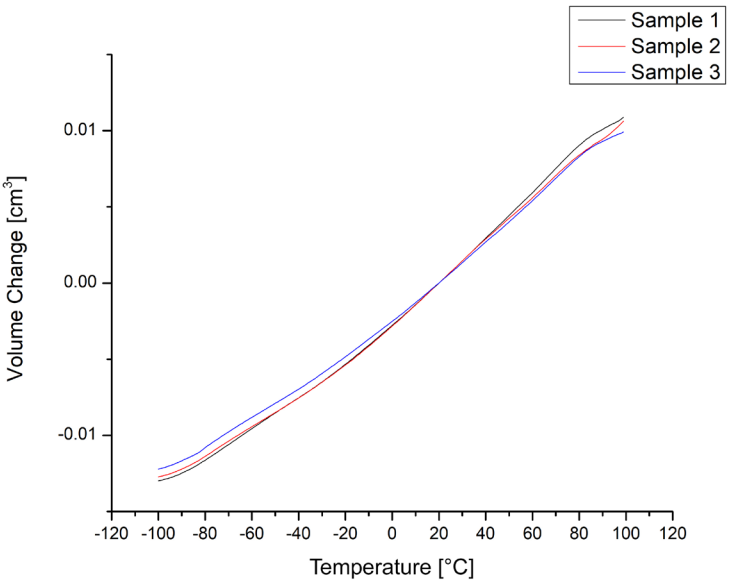


Figure 7. Variation of volume change of pellet with temperature

4.4 Density vs temperature

The density of explosives is directly related to their detonation velocity, which is the decisive parameter for all types of explosives. The density of the material is directly related to the thermal properties of the explosive. A material with a higher density has a higher value for CTE, thermal expansion, *etc*. A study of the density of PBX-9502 as a function of temperature was discussed in the range from –50 to +150 °C [18] and theoretical modelling was predicted based on volumetric thermal expansion [21]. In the present work, three samples with an initial material density of 1.57, 1.50 and 1.43 g/cm^3 were subjected to thermal heating in the range from –100 to +100 °C. A change in densities was observed over a wide temperature range and the densities were plotted against temperature. The change in volume of the explosive leads to a change in density. The pattern of density change over the entire temperature range was the same for all three samples. The observed slope was different in the range from –100 to –20°C to that above 20 °C. The rate of density change is lower in the lower temperature range than above 20 °C. Below ambient temperature, the explosive shrinks overall, resulting in a reduction in volume while the mass remains the same; therefore, the density increases monotone and vice versa. These results indicated an almost linear variation of the densities in the range from –100 to +100 °C.

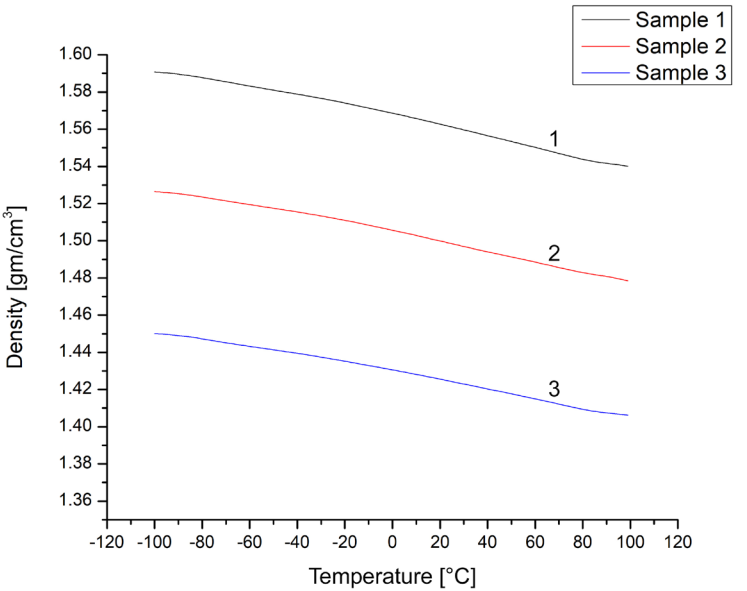


Figure 8. Variation of density of pellet with temperature

4.5 Irreversible growth

To ensure the repeatability of the data, each sample was subjected to three test runs. After analyzing the data, it can be seen that the CTE value decreases after each test run over the entire temperature range. This irreversible growth of the explosive material is referred to as ratchet growth. The reason for the change in CTE values during thermal cycling for RDX/wax (95/5) is not known, but a similar pattern is described as irreversible thermal expansion in energetic materials [8]. Woznick *et al.* [16] reported the increase in specific volume and dilatancy with repeated thermal cycling. The same phenomenon is also observed with PBX-9502 and other TATB-based compositions [7, 8, 12, 16, 17, 21, 22]. The deviation of the CTE values of the RDX/wax (95/5) samples between the 2nd and 1st, and the 3rd and 2nd test runs, was calculated as a percentage change. The values for the two samples are listed in Table 3.

Table 3. CTE values at different temperatures after thermal loading

	CTE value ($\times 10^{-6}$/K) in temperature [°C]						
	–80	–60	–40	0	40	60	80
Sample 1/4							
Test Run 1	52.578	53.741	56.495	61.293	65.984	68.858	71.621
Test Run 2	51.835	52.809	55.401	61.186	65.562	67.778	70.447
Test Run 3	51.292	52.192	54.818	59.935	65.075	66.630	69.455
Change 1-2 [%]	1.413	1.735	1.937	0.175	0.640	1.570	1.640
Change 2-3 [%]	1.047	1.166	1.052	2.045	0.744	1.693	1.408
Sample 2/4							
Test Run 1	54.879	55.672	58.167	62.084	61.451	59.296	61.183
Test Run 2	54.794	55.894	56.587	61.4166	60.031	56.758	58.638
Test Run 3	54.597	55.649	57.483	61.6131	59.435	55.345	57.195
Change 1-2 [%]	0.154	–0.399	2.716	1.076	2.309	4.281	4.160
Change 2-3 [%]	0.360	0.439	–1.584	–0.320	0.994	2.487	2.461

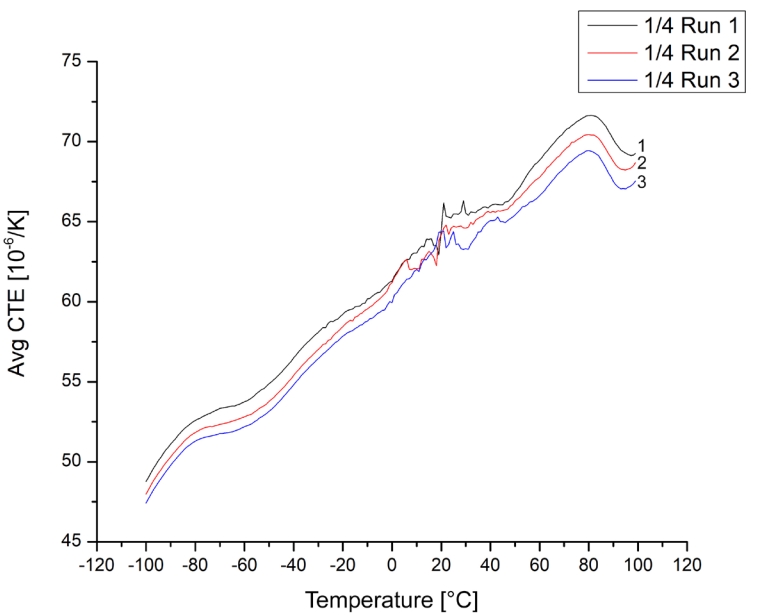


Figure 9. CTE values on thermal cycling

On analyzing these values, it was observed that the percentage changes between two thermal cycles lies within the range of 0.2-4%. The reason for this change is not well established for RDX/wax explosives. However, the variation

may be due to irreversible thermal expansion or ratchet growth. This cause cannot be totally ruled out as the reason for ratchet growth is due to the interaction of energetic crystals with each other and with the binder, as has been established for other classes of explosives.

5 Conclusions

- The thermal behaviour of RDX/wax (95/5) composition explosive pellets was investigated using Thermo Mechanical Analyzer (TMA), Differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), and Thermal Property Analyzer (TPA) techniques. The explosive composition was investigated in a wide temperature range of –100 to +100 °C using TMA techniques.
- Pressed RDX-wax explosives are one of the most used types of explosive and thermal expansion data in the temperature range of –100 to +100 °C will be useful for predicting the behaviour of these explosives under different thermal conditions.
- This investigation may also be useful in the study of structural health monitoring of warheads and explosives.
- Irreversible thermal expansion was observed for the RDX/wax (95/5) explosive composition; however, to validate ratchet growth further, more thermal cycling will be required.

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Contribution

Vijay Pratap Mall: conception, foundations, methods, performing the statistical analysis
Nilesh Naik: conception, foundations, methods, other contribution to the publication
Rakesh Kalal: conception, foundations, other contribution to the publication
Sangeeta Kale: foundations, performing the experimental part, other contribution to the publication

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