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Thermogravimetric study of vapor pressure of TATP synthesized without recrystallization

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ABSTRACT

This study aims at characterizing the vapor pressure signatures generated by triacetone triperoxide (TATP) that was synthesized without recrystallization by thermogravimmetric analysis (TGA) for exploitation by standoff detection technologies of explosive devices. The thermal behavior of the nonrecrystallized sample was compared with reported values. Any phase change, melting point and decomposition identification were studied by differential scanning calorimeter. Vapor pressures were estimated by the Langmuir method of evaporation from an open surface in a vacuum. Vapor pressures of TATP at different temperatures were calculated using the linear logarithmic relationship obtained from benzoic acid reference standard. Sublimation of TATP was found to follow apparent zero-order kinetics and sublimes at steady rates at 298 K and above. While the enthalpy of sublimation found, 71.7 kJ mol^{-1}, is in agreement with reported values the vapor pressures deviated significantly. The differences in the vapor pressures behavior are attributable to the synthesis pathway chosen in this study.

1. Introduction

Improvised explosive devices (IEDs) are a common and growing threat to human populace world over. Detecting and identifying explosives charges and IEDs are very arduous with increasing risks associated with the task. The diverse and varying nature of IEDs poses great challenges to the methods for their detection. In order to set requirements and specifications for detection equipment and to design suitable, capable, and robust detection systems, it is pertinent to know the properties of the species to be detected.

One property of vital importance for detection methods based on trace detection, such as sniffers or stand-off vapor detection techniques is the vapor pressure. Also of equal importance is the spectral signature of TATP. A high vapor pressure ensures an increased probability of finding a hidden or partially enclosed explosive (or a fully enclosed explosive, encapsulated in a gas permeable casing). The enthalpy of sublimation or enthalpy of vaporization is also of importance for the modeling of explosives vapor plumes from IEDs and is determined by measuring the vapor pressure as a function of temperature [1]. A large focus has been geared to collecting a data base that contains sublimation and vaporization enthalpies data of many explosives [2].

A widely used laboratory approach to obtain ambient vapor pressures is gas chromatography. Advantage is taken of the increased vapor pressures of materials with elevated temperature. The temperatures and vapor pressures can then be fitted to the Clausius–Clapeyron equation allowing for extrapolation of vapor pressures at other temperatures [3–5]. Good linearity for this equation implies that the enthalpy of sublimation is relatively constant over the temperature range.

Another route, thermal gravimetric analysis (TGA), is used in this study to obtain vapor pressures and heat of sublimation of TATP. One application that extensively uses the TGA method for estimation of vapor pressures of materials is the pharmaceutical industry [6–9].

Our aim is to compare the impact of synthesis without recrystallization to the common synthesis pathways, which utilize recrystallization step, on the thermal influence of TATP in order to develop some important vapor signatures that can be exploited in designing its detection remotely or using standoff capabilities. TATP samples used in this study were synthesized in small batches by licensed personnel in the laboratory at the Naval Surface Warfare Center. The thermogravimetric study of TATP synthesized without recrystallization has not been reported elsewhere. As a result, the vapor pressures as investigated herein reflect actual values in real life application, which avoids recrystallization step. Thus, we report on the vapor pressure of TATP synthesized

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without recrystallization, and its enthalpy of sublimation as determined by a TGA.

2. Material and methodology

Triacetone triperoxide (TATP) was synthesized in our laboratory based upon works of previous researchers [3,10] without the recrystallization step, and benzoic acid standard was purchased from Sigma-Aldrich. Since the scope of this effort was to study TATP similar to what is made in clandestine laboratories, the product was not recrystallized. The recrystallization step would be used to remove contaminants and any trapped acid that remains after the synthesis. It should be noted that since this step was not performed, this may have impact on the vapor pressures of the material studied and may also impact the calculated enthalpy of sublimation. Thus, this study reports the results of TATP synthesized without recrystallization.

All TGA analyses were performed on the TA Instruments Q500 Thermogravimetric Analyzer after calibration with Curie point standards for temperature at 5 K/min. The samples were analyzed using nitrogen as the purge gas at the manufacturer-suggested rate of 1.0×10^{-6} m^3/s flow for the furnace and a 6.7×10^{-7} m^3/s flow for the balance housing. Platinum TGA pans containing 5 mm I.D. open aluminum pans (to hold the TATP samples) were used for all analyses. An open aluminum pan of similar mass to those used for samples was also placed in the balance housing prior to calibration of the instrument. TGA sample mass ranged from 2.0×10^{-6} to 5.0×10^{-6} kg but it was found the use of 5.0×10^{-6} kg was a better choice as TATP was less likely to be fully consumed during the analysis.

Dynamic TGA and derivative thermogravimetric (TGA-DTG) were performed for kinetic study. TGA measures the amount and rate of mass change of a material as a function of temperature or time in a controlled atmosphere. The technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration. We have used this technique to find the vapor pressures of TATP and its enthalpy of sublimation. The Langmuir evaporation in a vacuum from TGA weight loss is used together with the Clausius–Clapeyron equation to estimate sublimation enthalpy. Two forms of Langmuir equations were used to obtain the vapor pressures of TATP and both compared: one using the direct equation and the other by pseudo Antoine constants.

All Differential Scanning Calorimetry (DSC) analyses were performed on the TA Instruments Q20 DSC after calibration at 1 K/min using an indium standard for temperature/heat flow and a lead standard for temperature. Nitrogen was used as the purge gas at the manufacturer's-suggested rate of 8.3×10^{-7} m^3/s. TA's aluminum hermitically sealed Tzero© pans were used for calibration and for all analyses. DSC analysis was used for initial characterization of newly synthesized TATP and to determine its melting point. The TATP sample mass used for the melting point was approximately 3×10^{-6} kg.

Vapor pressure estimation was carried out by the Langmuir approach. This is based on the determination of the rate of evaporation of the sample from an open surface in a vacuum. The value of α is constant if the initial conditions were vacuum, but did not exclude the condition that it may have different values. When in vacuum, α is regarded to be unity, and in flowing gas atmosphere as found in TGA experiments α takes a significantly different value. However, for the application of the Langmuir expression, α is a constant even in the purge gas [9]. The vapor and the condensed phases are in dynamic equilibrium. Eq. 1 is developed for Langmuir evaporation from an open surface using a TGA instrument which was initially derived for vacuum [11]. One important assumption for the Langmuir equation to be valid as used in TGA experiments is that the mass loss must occur from a given container exposing a constant area of interface.

$$G = \alpha p^* \left(\frac{M}{2\pi RT}\right)^{1/2} \tag{1}$$

where T is the temperature in K, R is the universal gas constant, α is a coefficient of evaporation or accommodation simply known as the Langmuir coefficient assuming that an obstacle to transfer from the condensed phase to the gas phase and returns exists. G is the mass per m^2 of surface which the substance losses per unit time for evaporation at temperature T in a vacuum, p^* is the vapor pressure, and M is the molecular weight of the specie.

From Eq. 1,

$$\text{let } c = (2\pi R)^{1/2}/\alpha \tag{2a}$$

$$\text{and } f = G(T/M)^{1/2} \tag{2b}$$

Therefore,

$$p^* = \frac{G(2\pi RT)^{1/2}}{\alpha(M)^{1/2}} \text{ or } p^* = fc \tag{2c}$$

Applying the Clausius–Clapeyron equation,

$$\ln p^* = -\frac{\Delta H_{sub}}{RT} + B \tag{3a}$$

and,

$$\ln(fc) = -\frac{\Delta H_{sub}}{RT} + B \tag{3b}$$

where ΔH_{sub} is the enthalpy of sublimation, R is the universal gas constant, B is a constant and T is the temperature in K.

Hence,

$$\ln f = -\frac{\Delta H_{sub}}{RT} + B - \ln c \tag{3c}$$

To determine the vapor pressure by the Langmuir method, it is necessary to measure the mass loss per unit surface per unit time, G. This can be accomplished by measuring the loss in weight of the sample using the thermogravimetric analyzer instrument, TGA and by applying Langmuir's model for evaporation in a vacuum to estimate vapor pressures and enthalpy with αnot equal to unity.

Also, studies have shown that the logarithm of the sublimation/evaporation rate at a given temperature is linearly proportional to the logarithm of vapor pressure [12,13]. Application of this concept suggests that the linear relationship between sublimation rate and vapor pressure is independent of the material used and the temperature range in which the experiments are carried out but is dependent on the specific instrumental system, experimental conditions, and sample containment procedure [14,15].

Benzoic acid has been recommended as a calibrant for enthalpy of sublimation measurements of substances having a vapor pressure of approximately 0.1 Pa at 298.15 K or 10–360 Pa from $338 < T$ (K) < 383; all available experimental evidence suggests that it is a suitable reference material when measured under these specified conditions [14–17]. The linear logarithmic relationship between the sublimation rate of benzoic acid and the corresponding vapor pressure is demonstrated by a plot of (f vs. p^*) from which Langmuir coefficient (α) is obtained. The vapor pressure of TATP is calculated from Eq. 2c and enthalpy of sublimation is obtained from a plot of ($\ln f$ vs. $1/T$) or from the Clausius–Clapeyron Eq. 3(a).

3. Results and discussion

3.1. Determination of the Langmuir coefficient, α

A set of experimental mass loss data, G, of benzoic acid without smoothing is shown in Fig. 1. The values of G obtained from these isothermal plots are used in Eq. 2(b) to calculate f. The vapor pressure data for benzoic acid were taken from the literature [13,14] and are plotted against f as shown in Fig. 2.

Measurements of the mass loss were performed at temperature ranges from 298 to 350 K at different temperature increments. Using the Langmuir Eq. 2(a), a value of α under the operating condition was calculated. Once the apparatus had been calibrated in this way for α, the vapor pressures of unknown materials could be measured. By using the appropriate value of α, calculated from the benzoic acid data obtained under a certain set of thermogravimetric conditions, the vapor pressure of TATP was calculated from the data obtained under these operating conditions.

3.2. Determination of vapor pressure of TATP

Fig. 3 shows a set of representative experimental time curves (TGA thermograms) depicting weight loss data of TATP from 298 to 318 K without smoothing at different temperature increments.

Determination of the sublimation rate of TATP requires the sample to be at least 95% pure and without substantial amount of residue solvent or other volatile impurities. From 298 to 309 K, weight loss plotted vs. time from the thermograms, were linear, which indicated that the sublimation followed apparent zero-order kinetics. Similarly, at temperatures above 309 K, the weight loss curves of TATP were also linear up to some point in the time range and followed apparent zero-order kinetics but became nonlinear afterwards, possibly due to, changes in surface area, and depletion of the crystals.

The enthalpy of sublimation generated from the vapor pressure data for TATP measured by thermogravimetry can be estimated from the Clausius–Clapeyron Eq. 3(a) or by Eq. 3(c) as given by plot of p^* or f vs. reciprocal temperature shown in Figs. 4 and 5. The enthalpy of sublimation, (ΔH_{sub}) obtained from Eqs.3(a) and 3 (c) are 71.7 and 74.6 kJ mol^{-1} respectively. The enthalpy measurements are in good agreement with literature data on this material [12]. Reported literature values of the sublimation enthalpies ranges from 72 to 109 kJ mol^{-1}, even though, recrystallization step was a part of those studies. The discrepancies noted in the literature sublimation enthalpies may have resulted from impurities and other oligomeric peroxides present in TATP crystal. In the light of the present study, there was no noticeable effect of TATP recrystallization and its enthalpy of sublimation. However, the vapor pressures obtained are not in agreement with the reported values as discussed next. Consequently, we have shown that by using the appropriate α, the vapor pressure can be obtained from the TGA data for the material under investigation.

The vapor pressure values and sublimation rates are presented in Table 1 at temperature range of 298–327 K. The temperature dependency of the sublimation rate and vapor pressures are evident. Thus, this method provides convenient route of estimating the vapor pressures of TATP at the temperature range of study. From the present work, we did not find any supporting evidence to conclude that recrystallization has any effect on the sublimation enthalpy of TATP based on the reported literature values since some of these values are in agreement with the value obtained in this study.

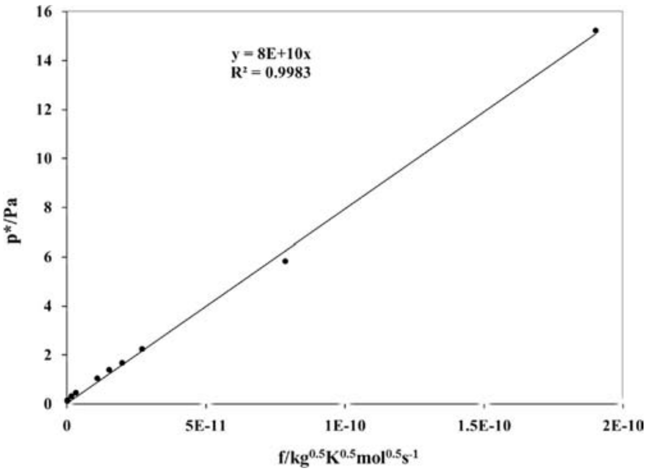

Fig. 2. Vapor pressure calibration using known benzoic acid vapor pressure at isothermal mass loss in the temperature range 298–350 K [12,13].

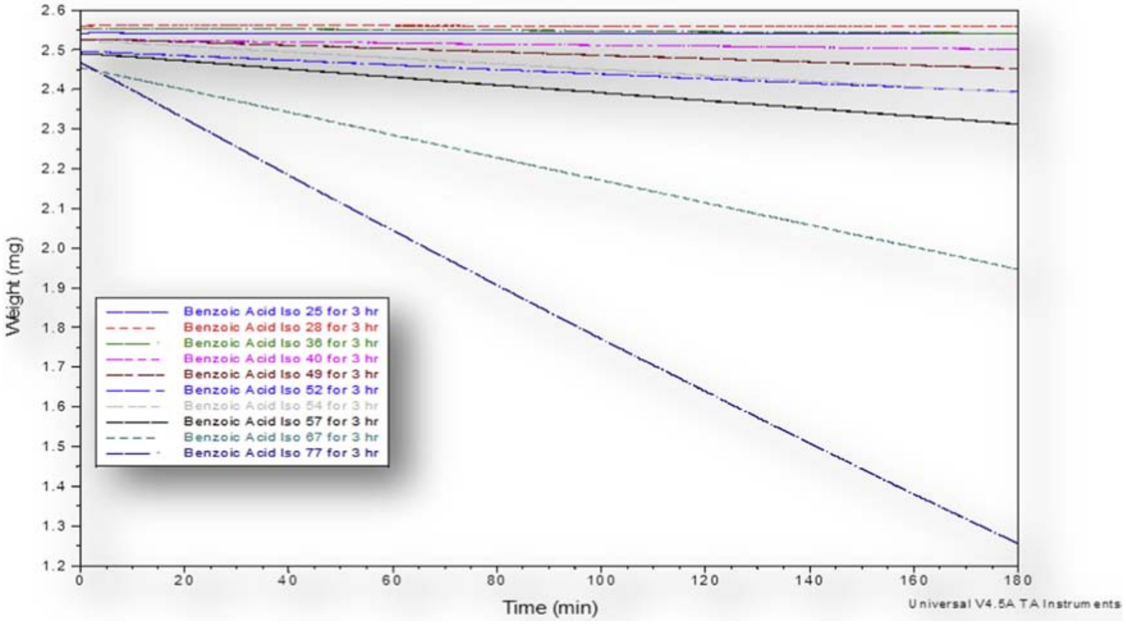

Fig. 1. The representative weight loss thermograms from isothermal thermogravimetric analysis of benzoic acid from 298 to 350 K after 3 h of operation.

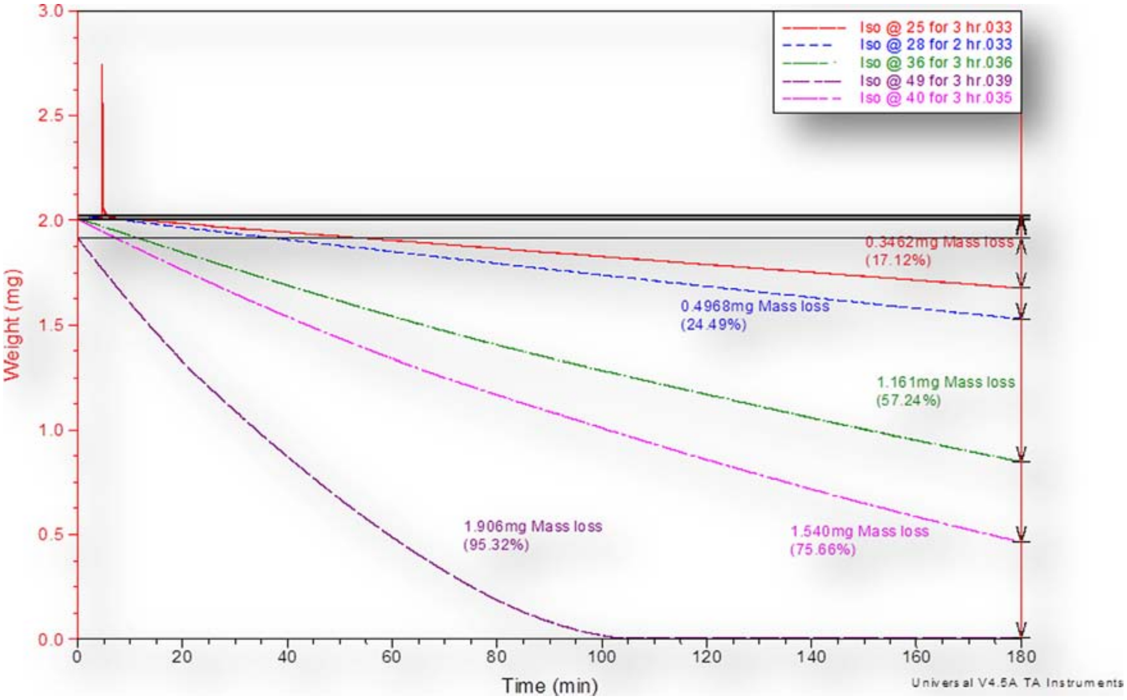

Fig. 3. The representative weight loss thermograms from isothermal thermogravimetric analysis of TATP from 298 to 322 K.

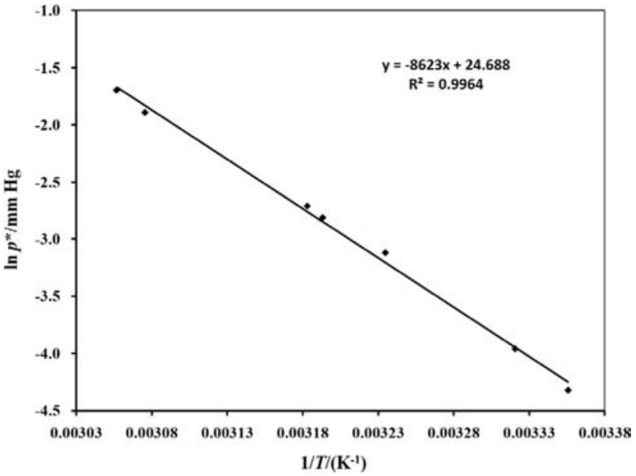

Fig. 4. Estimation of TATP enthalpy of sublimation from Clausius–Clapeyron Eq. 3a for isothermal TGA.

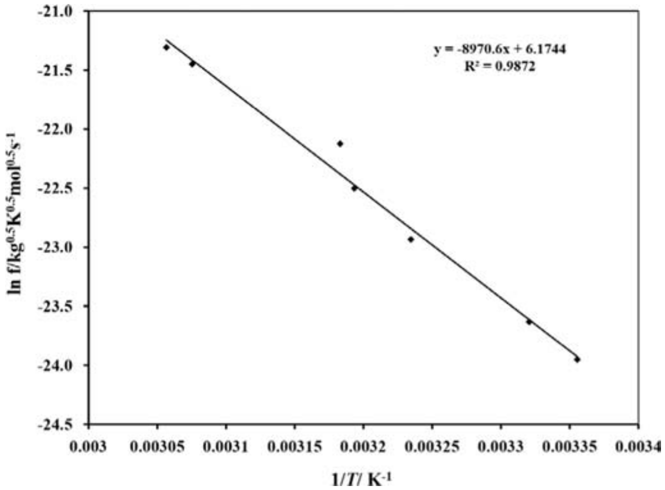

Fig. 5. Estimation of the enthalpy of sublimation of TATP from the Langmuir model, Eq. 3(c) by isothermal TGA.

Vapor pressures were compared to NIST data archives of phase boundary pressure for crystals as shown in Fig. 6. The vapor pressure data obtained is inconsistent with reported values at temperature range, nevertheless, the enthalpy of sublimation falls within the reported values. A possible explanation for this deviation is probably due to the increased number of molecules of TATP oligomers and carryover materials in the nonrecrystallized sample. Oligomers may include diacetone diperoxide (DADP), cyclic dimers and tetramers, monomers, etc., and have been reported to affect the stability of TATP. The acid-catalyzed peroxidation of acetone leads to up to three forms of acetone peroxide, including a monomer, cyclic dimer, and a cyclic trimer [18]. The trimer is generally thought to be marginally more stable than the other two and is formed preferentially when the temperature of the reaction and subsequent product formation is kept below 283 K and may also be induced by other factors. Some of the more unstable dimers are always found associated with the trimer. As a result, the calculated vapor pressures are substantially minimized compared to reported values that utilized recrystallization in the synthesis pathway. This result is significant given that a high vapor pressure ensures an increased certainty of finding a hidden or partially enclosed explosive (or a fully enclosed explosive, encapsulated in a gas permeable casing). Note that the temperature range of study falls within the applicable temperatures during

Table 1
TATP sublimation rate, G from 298 to 327 K and the estimated vapor pressures, p^*.

T (K)	Sublimation rate, G (kg s^{-1}) ($\times 10^{11}$)	Langmuir expression, p^* (Pa)
298	3.20	1.73
301	4.60	2.49
309	10.74	5.81
313	14.26	7.71
322	17.65	9.54
325	36.41	19.68
327	45.04	24.35

regular routine screenings at the airports, seaports, and other locations of interest. However, detail analyses of any oligomers and/or carryover materials that may be present in the samples are beyond the scope of this study. It must also be noted that the only difference between our synthesis pathway and that of other authors whose works are compared to in this study is the avoidance of recrystallization step.

3.3. *Kinetics evaluation*

As a substance undergoes phase transition from solid to vapor, determining the rate of mass loss, *G*, can monitor the sublimation process. This is achieved by thermogravimetry, using a linear rising temperature program. The TGA curve that shows a plot of mass against temperature or time is converted into a DTG curve, which measures the rate of mass loss. In the present study, the sublimation of TATP takes place at the solid–gas interface. Triacetone triperoxide synthesized in our laboratory is crystalline with a melting point of 359 K as determined by DSC. The decomposition temperature is near 473 K. Mass changes that arise during thermal analysis using TGA and the DSC data are an indication of the stability of TATP within the thermal range of study. Fig. 7 is an overlay of TGA and DSC thermograms obtained in a dynamic study at 5 K min^{-1} (TGA) and 1 K min^{-1} (DSC) heating rate. There is a high loss of mass before the phase transition temperature (melting point). The sharp transition at 367 K is present in all the experiments conducted and may be related to the melting point since many literatures have reported on different melting points. Nevertheless, the weight loss observed occurred before any of the temperatures at 359 and 367 K which, supports the conclusion reached.

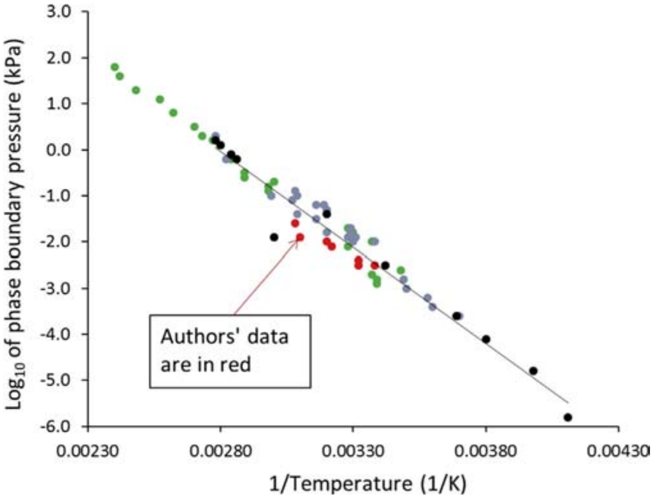

Fig. 6. Comparison of authors' vapor pressures data to literature from NIST Data Archive [19–24].

Since this temperature range was below the melting point of TATP, the weight loss was actually due to sublimation. Therefore, TATP can be used for thermogravimetric study of energetic materials due to its mass loss below the melting point.

Two endothermic peaks are evident in the melting point range of TATP. The first endotherm has an average temperature of 359.95 ± 273.05 K with an average observed enthalpy of fusion 26.3 ± 7.9 kJ/kg. The second endothermic peak yielded an average melting point of 367.85 ± 273.05 K with an average observed enthalpy of fusion 114.4 ± 6.3 kJ/kg. This phenomenon is likely due to the presence of a carryover materials or oligomer in TATP matrix.

Data obtained from the TGA curve are converted into a DTG plot to obtain the rate of mass loss with temperature as shown in Fig. 8. For this process to be zero order in nature, the DTG plot of the % mass loss per unit time will have a maximum peak value for the rate of mass loss at the point where the mass of the material is enervated. This would cause an abrupt return of the rate of mass loss curve to a zero baseline. Experiments carried out in this study had all the characteristics as described above. Thus, it was assumed that all the isothermal analyses are undergoing zero-order kinetics.

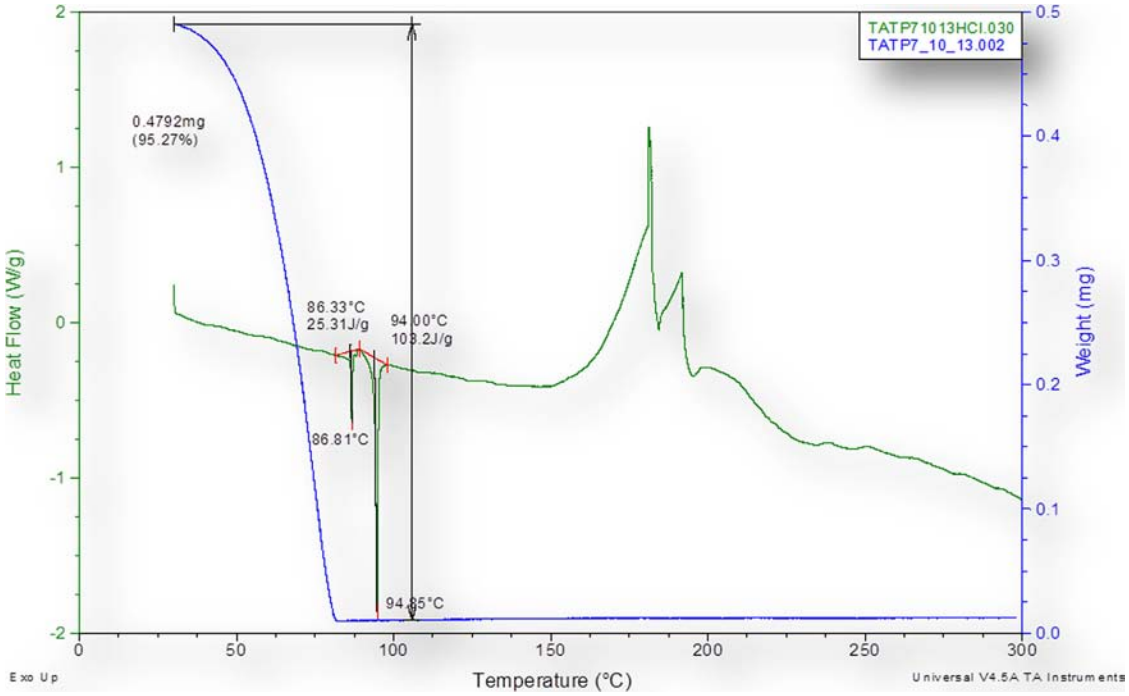

Fig. 7. TATP heat flow (primary axis) at 1 K min^{-1} and sublimation rate (secondary axis) at 5 K min^{-1}. The high mass loss before melting point is an indication of sublimation and not vaporization.

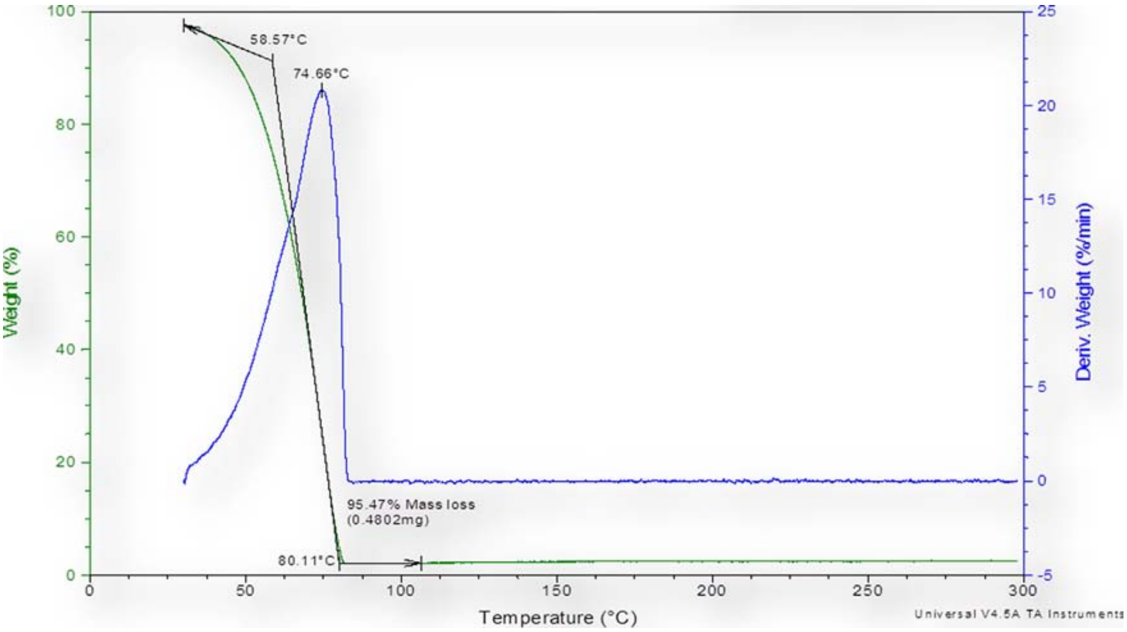

Fig. 8. TGA-DTG plot for TATP showing zero-order kinetics.

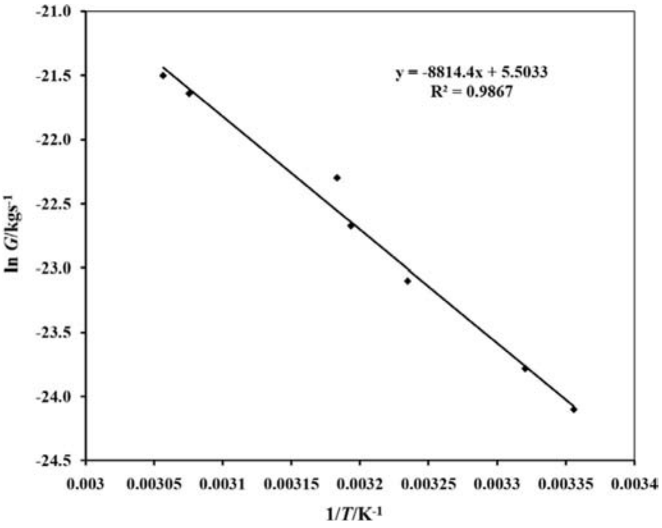

Fig. 9. Activation energy of TATP from isothermal mass loss rate data.

Eq. 5 is an Arrhenius relationship used to obtain the activation energy (E_{act}) of TATP.

$$G = Ae^{\frac{-E_{act}}{RT}} \qquad (5)$$

Fig. 9 is a plot of mass loss rate, G vs. temperature using Eq. 5, and from which the E_{act} is evaluated from the slope. A value of 73.3 kJ mol^{-1} is obtained for E_{act}. This value differed by 1.6 and 1.3 kJ mol^{-1} from the values obtained for ΔH_{sub} estimated using Eqs. 3(a) and 3(c) respectively. In all non-activated sublimation kinetics, the E_{act} and ΔH_{sub} have very close values. Thus, the enthalpy of sublimation obtained using thermogravimetry is in good agreements with its activation energy.

3.4. Gas chromatograph mass spectrometry (GC/MS) analysis

Fig. 10 is the chromatogram of TATP using a GC–MS instrument. Analyses of all TATP samples were conducted on a Hewlett Packard HP 6890 gas chromatograph coupled to a HP 5973 mass spectrometer. A HP-5MS (30 m × 0.25 mm × 0.25 μm) capillary column was utilized in the GC. Helium carrier gas was maintained at 8.0×10^{-8} m^3 s^{-1} throughout all analyses. The GC injector port was held at 438.15 K with splitless injection during all data collection runs.

The chromatogram contained a single prominent TATP peak with an abundance of approximately 700,000 counts or $>95\%$ composition based on a 2.0×10^{-9} m^3 syringe injection. The chromatographic responses of TATP in all analyses were in order of magnitude higher than carryover compound in the matrix. Thus, the low TATP vapor pressures calculated from the TGA analysis compared to literature reported values may have resulted mainly from oligomeric materials in addition to carryover materials in TATP matrix. Analysis of these constituent materials is beyond the scope of this work. As a result, the net effect is a reduction in the number of TATP molecules contained in the vapor phase which consequently impacted the vapor pressures. As mentioned previously, synthesis pathway with recrystallization would likely contain less of these oligomers and/or impurities than the pathway without recrystallization.

3.5. Raman spectroscopy and Fourier transform infrared spectroscopy (FT-IR) analyses

Raman spectra were collected using a Thermo Scientific NXR FT-Raman module connected to a Nicolet 6700 FT-IR. All spectra reflect 64 sample scans. Spectra were collected from TATP contained in 5.0×10^{-6} m^3 vials. A typical Raman spectrum of TATP synthesized using HCl and H_2SO_4 as the catalysts is shown in Fig. 11. There is no notable difference between the two materials. This spectrum exhibits the Raman vibration frequencies typical of TATP. The large peaks at 2947 and 3011 cm^{-1} are due to CH_3 asymmetric stretching while the peaks between 850 and 1000 cm^{-1} are assigned to the peroxide O–O stretch. Specifically, the peak at 946 cm^{-1} has been shown to be associated with asymmetric stretching of the O–O bond within TATP. The low-frequency bands near 550–600 cm^{-1} are associated with the O–C–O bending motion [10,25].

Further TATP identification was conducted with FT-IR using a Thermo Scientific Nicolet 6700 FT-IR with a diamond attenuated total reflectance (ATR) sampling cell. All spectra reflect 32 sample

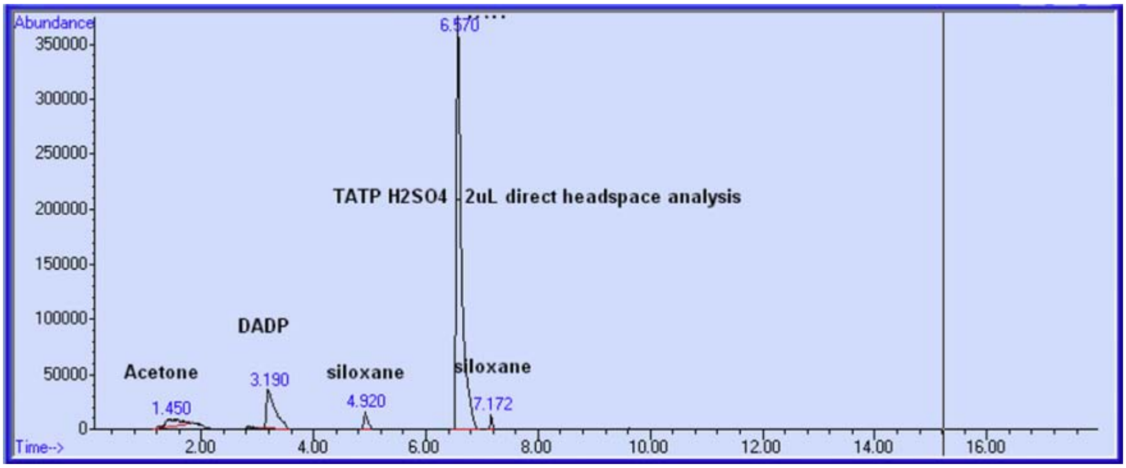

Fig. 10. Chromatogram of TATP showing the presence of material carryover from synthesis.

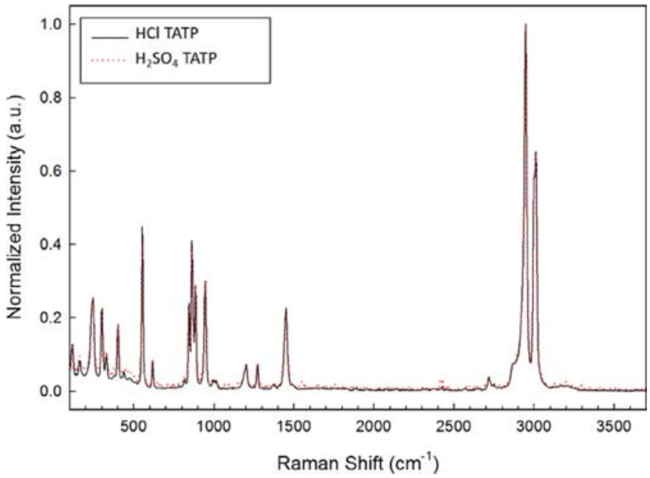

Fig. 11. Raman spectrum of TATP synthesized using HCl and H_2SO_4 as the catalysts.

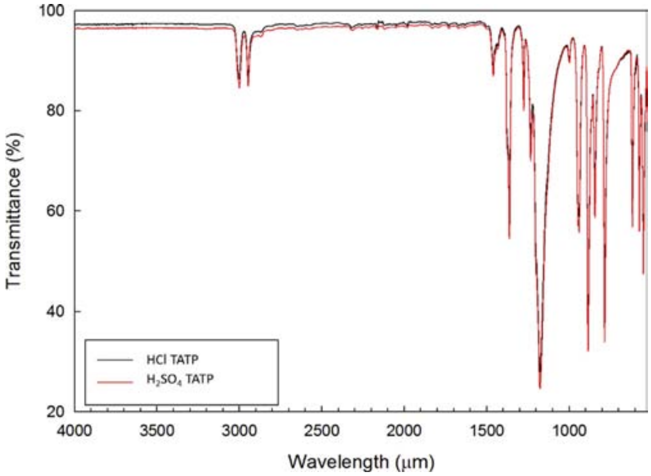

Fig. 12. Normalized FT-IR spectrum comparison of HCl and H_2SO_4 catalyzed TATP.

scans. FT-IR spectra of TATP synthesized using HCl and H_2SO_4 as catalysts are shown in Fig. 12. These spectra show several large absorbance peaks that are indicative of peroxide compounds. Specifically, the absorbance at 1177 cm^{-1} reflects the C–O bond stretching and the absorbance values between 784 and 938 cm^{-1} reflect O–O bond stretching. This spectrum was consistent in all TATP syntheses and reflects expected behavior of a peroxide based material. There is no significant difference between the two samples.

4. Conclusions

The vapor pressures and enthalpy of sublimation of un-recrystallized, high energetic explosive, TATP, were successfully determined by thermogravimetric analyses. Vapor pressure is a strong indicator for detection technology hence, the result achieved in this study may become a safety standard for trace or ultra-trace detection. The enthalpy of sublimation obtained, 71.7 kJ mol^{-1}, from Clausius–Clapeyron equation using benzoic acid reference standard to derive a linear logarithmic relationship between sublimation rate and vapor pressure specific to the TGA system is consistent with reported values. However, the vapor pressures deviated substantially from the reported values. The difference in the vapor pressures behavior can be attributed to the synthesis pathway chosen in this study. TATP undergoes sublimation at steady rates at 298 K and above, following apparent zero-order kinetics. In light of this study, the results obtained can be tailored effectively for exploitation by TATP detection technologies. This work also demonstrates the effectiveness of thermogravimetry for the determination of un-recrystallized TATP vapor pressures.

Acknowledgments

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