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Stability and hazard properties of improvised nitrocellulose

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ABSTRACT

Nitrocellulose is a vulnerable and sensitive energetic material that can easily be manufactured from ordinary household chemicals and used for illicit purposes. Hazards of this material can lead to incidents resulting from unplanned initiation in manufacturing, storage or handling.Thermal stability and hazard data for so-called improvised nitrocellulose (IN) are crucial to formulate guidance for storage and handling of such materials recovered from illegal possession. This study focused on the energy content, stability and safety properties of IN. The energy contents increase linearly with nitrogen contents of IN. Improvised nitrocellulose showed a similar sensitivity to impact and electrostatic discharge, but vacuum stability testing demonstrated more gas release (2.8–3.4 $cm^3\ g^{-1}$) compared to commercial NC (1.7 $cm^3\ g^{-1}$). Chemiluminescence analysis established activation energies for hydrolytic and thermolytic decomposition to be 36 and ~93 kJ mol-1, respectively. Results suggest that NC samples recovered from improvised devices or illegal possession should be handled with special care.

KEYWORDS

Chemiluminescence; decomposition; improvised; sensitivity; stability

Introduction

Organic energetic materials such as nitrate esters are extensively used in military warheads and mining applications. Nitrocellulose is a nitrate ester of cellulose and its applications are determined by the degree of nitration. The nitrogen contents for pure mono-, di- and tri-substituted nitrate esters are 6.76%, 11.11% and 14.14%. The latter is a theoretical maximum, and cannot be achieved. 13.8% is taken as the highest attainable nitrogen content which is in part due to the impenetrable crystalline regions of the natural cellulose (Fernández de la Ossa et al. 2011). Commercial nitrocellulose with a nitrogen content greater than 12.6% is primarily used for the manufacture of explosives, whereas variants with a lower nitrogen content are used as additives in products such as paints, lacquers and printing inks (McIntyre and Price 1981; Stoffel 1968). Two major forms of nitrocellulose are used in explosives: grade A (pyrocellulose) with a nitrogen content of 12.6% is used in propellants, and grade B (gun cotton) with a nitrogen content of 13.35–13.45%

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is used in both propellants and as a primer in electric detonators. The nitrogen content of commercial nitrocellulose determines its chemical, physical and ballistic properties and is one of its most important attributes.

Nitrate esters undergo a number of age-related decomposition processes. The intrinsic decomposition involves the loss of NO_2 by hydrolytic cleavage of nitrate ester bonds of CO-NO_2, due to its smaller activation energy (below 150 kJ mol^{-1}) (Brill and Gongwer 1997). This process produces alkoxy and NO_2 radicals. Other decomposition involves the thermolytic decomposition at above 60° C having an activation energy above 150 kJ mol^{-1}. If the stabilizer level reaches below the threshold level (below 20%) the NOx reacts with moisture and produces acid. This acid then accelerates the decomposition reaction by a process called auto-catalytic reaction, leading to autoignition of the NC or NC based propellants. All secondary and subsequent decomposition reactions are resultant from intra- and intermolecular reactions of these radicals. The kinetics and decomposition mechanisms of these decomposition processes have been extensively studied (Moniruzzaman, Bellerby, and Bohn 2014; Tarchoun et al. 2021, 2022).

Commercial nitrocellulose is a high-quality product manufactured by nitrating pure cotton linters or wood cellulose primarily by sulfuric acid and nitric acid mixture. Its physical characteristics differ from white fibers to thick liquid. The use of nitrocellulose is not limited to making smokeless gun powder, rocket motors, waterproof fuses in pyrotechnics, it is also used in inks, adhesives, varnishes, resins, lacquer coatings, embedding sections in microscopy, photography, electrotechniques, galvanoplasty, and even certain plastics etc.

There are many easily accessible information sources on the Internet showing how to produce improvised forms of nitrocellulose from household products such as drain cleaning acids and potassium nitrate. Therefore, an improvised nitrocellulose is the nitrated product of crude cellulose from shops, newspapers, denims etc. The cellulose is mainly nitrated using easily accessible nitrating agents such as KNO_3 and drain cleaning acid containing more than 95% sulfuric acid.

As one of the main uses of nitrocellulose is as a component of propellants and explosive formulations, the improvised nitrocellulose can be combined with simple initiation mechanisms to produce improvised explosive devices (IEDs) (Kolesar et al. 2013). However, the energetic properties of improvised nitrocellulose are largely unknown, and more information is needed to ensure the safe handling, storage and destruction of such materials. The safe storage and handling of nitrocellulose depend on its stability and shelf life because the decomposition can cause the release of gases and heat (Moniruzzaman, Bellerby, and Mai 2011). It is well documented that nitrocellulose undergoes slow decomposition even at sub-zero temperature due to lower activation energy of nitrate ester bond (O-NO_2). This degradation reaction releases

NOx gases which immediately react with moisture producing nitric acid. The acid generate heat and thus accelerates the decomposition reaction via autocatalysis (He et al. 2017; Mattar et al. 2020; Wei et al. 2019). This uncontrolled reaction ultimately can cause autoignition of propellants or NC stockpiles. This autocatalytic reaction is stopped by adding stabilizer such as secondary aromatic amines such as DPA, 2-NDPA or urea derivative such as ethyl centralite, methyl centralite etc, in the propellants (Luo et al. 2020). Although a great details of stability of commercial grade and military grade NC have been well investigated (Trache and Tachoun 2018), no stability and hazard data of improvised NC are available in the literature. A detailed understanding of such attributes is necessary for the safe management of stockpiles.

To address the lack of information about the stability of improvised nitrocellulose and its thermal, energetic and hazard properties compared to commercial-grade nitrocellulose, we prepared a common formulation using drain cleaning acid and potassium nitrate. The thermal decomposition and energy release was tested for a series of samples, and hazard properties such as impact and friction sensitivity, ignition characteristics and electrostatic discharge were evaluated. Vacuum stability and chemiluminescence data were also compared to a commercial-grade nitrocellulose sample. These data will not only help to predict the damage caused by IEDs but also highlight the circumstances that may trigger accidental ignition of nitrocellulose of unknown history, which will allow the development of guidance measures for the safe recovery of improvised nitrocellulose by law-enforcement and security personnel.

Experimental

Materials

All the reagents used in this study were commercially available household products. Cotton wool was purchased from Sainsbury's supermarket. Potassium nitrate, Hexeal drain cleaner (96% concentrated H_2SO_4) and One Shot drain cleaner (91% concentrated H_2SO_4) were purchased from Amazon online and were used to nitrate the cotton wool without further purification. The nitrogen content of the materials was determined by titration according to STANAG 4178 Ed. 2. The ferrous sulfate solution was prepared according to MIL-STD-286 method 209.11.1 (1991) with modifications to accommodate the use of 250 ml H_2SO_4 and manual titration. The solutions of H_2SO_4 and KNO_3, or H_2SO_4 and nitrocellulose, were transferred to a conical flask using a 50-ml pipette. A series of nitrocellulose samples was produced and samples with different nitrogen contents were tested and evaluated. The equivalent factor (Eq. 1) was used to calculate the nitrogen content (Eq. 2). The equivalent

factor was calculated using 2.46 g of KNO_3 based on the volumes and weights measured from the ferrous sulfate titrations:

$$F = \frac{W_{KNO_3} \times 0.13855}{V_{FeSO4}} \quad (1)$$

where F is the equivalent factor, W_{KNO3} is the weight of KNO_3, and V_{FeSO4} is the volume of titrant used for the calibration sample.

$$\%Nitrogen = \frac{V_{FeSO_4} \times F}{W_{NC}} \quad (2)$$

Where, V_{FeSO4} is the volume of titrant used for the nitrocellulose samples, F is the equivalent factor and W_{NC} is the weight of the nitrocellulose. The compositions of all the NC studied in this paper are listed in Table 1.

Thermal Analysis

The thermal decomposition behavior of the improvised nitrocellulose was investigated by DSC using the Mettler Toledo DSC 30 or DSC 822^e instruments. Samples (1.5–2.0 mg) were sealed in a pierced aluminum pan for testing under a nitrogen gas atmosphere. Measurements were taken over the temperature range 30–300°C at a heating rate of 10°C min^{-1}. Samples were tested in triplicate and the average results were calculated.

Explosive Hazard Testing

The nitrogen contents of samples JB5 and JB6 were very similar, so the hazard tests were conducted using a blend of these samples to determine the safety, performance, and likelihood of accidental initiation of the improvised nitrocellulose compared to the commercial reference sample (JBC).

Rotter Impact Test. The Rotter impact test was conducted in accordance with EMTAP test guidelines (2007) and the UN Transport of Dangerous Goods Manual of Tests and Criteria (2011). A 5 kg weight was dropped on 55 individual 30 mm^3 samples. A go/no go response for initiation was recorded and the height of the drop weight was increased or lowered depending on the response. The Bruceton staircase procedure was used to establish the median drop height for the improvised nitrocellulose. The figure of insensitiveness

Table 1. NC coding and their compositions.

Sample Code	Nitrogen Contents (%)
F	9.14
2A	9.81
JB2	12.80
JB4	12.61
JB5	12.65
JB6	12.53
JBC	12.6

(FoI) was then calculated in relation to the RDX standard, defined using Equation 3.

$$FoI = \frac{80H_{50}}{RM} \tag{3}$$

where 80 is, by definition, the FoI of standard RDX, H50 is the median drop height for the improvised nitrocellulose, and RM is the current value for the running mean drop height for improvised nitrocellulose.

Rotary Friction Test. The rotary friction test was used to establish the sensitiveness group of improvised nitrocellulose according to EMTAP test guidelines (2007). A 15 mm^3 (3 g) sample of nitrocellulose was spread onto a steel block and struck using a fly wheel in the Rotary Friction machine A "go/no go" response for initiation was recorded.

Temperature of Ignition Test. The temperature of ignition test was conducted as previously described (2011). Two 200 mg nitrocellulose samples in test tubes were placed into a block and heated at a rate of 5°C min^1. The temperature of ignition was recorded by the visual detection of flame, smoke or explosion.

Electrostatic Discharge Test. The electrostatic discharge (ESD) test was conducted as previously described (2011). A strip of polythene was prepared containing eight holes, each with a diameter of 6.35 mm and a depth of 3.2 mm. A strip of conductive self-adhesive copper foil was placed on one side of the holes to form the bottom electrode. The holes were then filled with nitrocellulose with gentle tamping and covered with another strip of self-adhesive copper foil to form the top electrode. The nitrocellulose samples were then subjected to discharges of 4.5, 0.45 or 0.045 J to determine their sensitivity.

Vacuum Stability

The vacuum stability test was carried out on the blended JB5/JB6 and a commercial grade NC containing 12.6% N sample (JBC) according to STANG 4556 () using the transducer method. Dried samples (2.5 g in each tube) were heated isothermally at 100°C for 40 h in three glass test tubes, and the evolved gases were monitored. The amount of gas was converted into cm^3 g^{-1} to measure the stability of the improvised nitrocellulose.

Chemiluminescence Analysis

Chemiluminescence is a technique that can be used to investigate the thermal decomposition kinetics of nitrate esters. This technique monitors the NOx gases from the decomposition of nitrate esters (Kimura 1989).

Chemiluminescence analysis was conducted using a Sievers NOA-280i nitric oxide analyzer. The carrier gas was nitrogen at a flow rate of 30 mL min^{-1}. The NO and NO_x ($NO + NO_2$) content of the carrier gas was analyzed using a high-sensitivity NO/NO_x chemiluminescence analyzer.

Results and Discussion

Thermal Analysis

The thermal properties of improvised nitrocellulose materials varying in nitrogen content were compared to the commercial reference JBC (Table 2). The improvised nitrocellulose samples were prepared by systematic variation of the nitration mixtures to achieve levels of nitration similar to those of commercial nitrocellulose. All improvised nitrocellulose samples were analyzed by DSC to determine the energy content, onset temperature and temperature of decomposition.

The decomposition temperatures of all the improvised nitrocellulose samples fell within the range 208–211°C which is in good agreement with the value reported for the decomposition of commercial nitrocellulose (190–210°C) (Sovizi et al. 2009). A decomposition temperature of 206°C was previously reported for nitrocellulose with a nitrogen content of 12.5% (Pourmortazavi et al. 2009). Our tests indicated that the decomposition temperature of JBC was 200°C which is within the range of values observed for the improvised nitrocellulose samples.

The combustion energy of the improvised nitrocellulose samples was determined by integrating the area of the exothermic peaks in the DSC traces and plotting them as a function of nitrogen content (Figure 1). The maximum energy content was~1910 J g^{-1} in sample JB2, with the highest nitrogen content of 12.8% tested in this study. This clearly shows that the enthalpy of decomposition rises almost linearly as the nitrogen content increases, confirming that the energy content of improvised nitrocellulose is largely dependent on the degree of nitration.

Table 2. Thermal analysis data for improvised nitrocellulose samples compared to the commercial reference sample JBC (obtained from Nitrochemie Wimims, Switzerland).

Sample Code	Onset temperature (°C)	Decomposition temperature (°C)	Average Energy (J g^{-1})
F	197	209	1110
2A	199	209	1408
JB2	199	211	1928
JB4	201	211	1703
JB5	198	208	1628
JB6	197	208	1676
JBC	200	200	1750

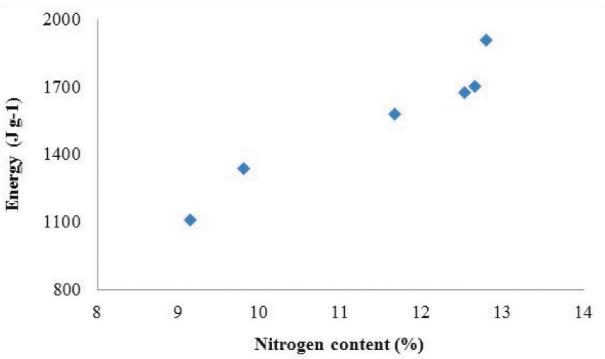


Figure 1. Plot of decomposition energy vs nitrogen content for nitrocellulose samples listed in Table 2.

Hazard Properties Analysis

Rotter Impact Test

Hazard property tests were conducted on the blend of samples JB5 and JB6 (Table 2). In the Rotter impact test, the ignition go/no-go results and volume of gas produced were recorded after 50 drops. The mean F of I for all tests was 22.8 with a standard deviation of 0.17, and a mean volume of 1.3 cm^3 of gas was produced for the go responses, in agreement with the F of I value of 23 reported for a commercial nitrocellulose with a nitrogen content of 13.4% (Börjesson and Westman 2015). The similar test results for improvised and commercial nitrocellulose indicate that both are sensitive to impact and similar initiation mechanisms are likely to be involved, i.e. the adiabatic heating of voids, suggesting that the void size and quantity must also be similar.

Rotary Friction Test

We conducted 10 tests at 158 rpm and no ignition was observed. This nil response equated to a figure of friction greater than 4.5, revealing that improvised nitrocellulose is not sensitive to friction, which is similar to the response of commercial nitrocellulose (JBC) containing 12.6% nitrogen. This was thought to be due to the decomposition of nitrocellulose before it had reached the ignition temperature.

Temperature of Ignition

Ignition temperature tests were carried out to assess the potential of improvised nitrocellulose for spontaneous ignition during progressive heating. The ignition temperature of the blended JB5/JB6 sample was confirmed to be 180°C, whereas the commercial nitrocellulose (JBC) ignited at 170°C (Judd 2016). The higher ignition temperature of the improvised nitrocellulose probably reflects the coating of some fibers by ammonium sulfate formed during

synthesis. Our data agree with other reported temperature of ignition values (169–185°C) (Thomas et al. 2011).

Electrostatic Discharge Test

The nitrocellulose was lightly tamped into a specimen holder and subjected to discharges of 4.5, 0.45 or 0.045 J. The improvised nitrocellulose was sensitive to an ESD of 4.5 J, but insensitive at 0.45 J. There was a clear ignition with an orange flash and mild audible output at 4.5 J. This suggests that the improvised nitrocellulose samples were not very sensitive to ESD (2007). The commercial nitrocellulose (JBC) also ignited in response to an ESD of 4.5 J but was not ignited at 0.45 or 0.045 J.

Vacuum Stability Test

The accelerated thermal aging of improvised NC was executed using a vacuum stability tester to determine the stability of NC based on measuring the gas liberated upon heating the NC. The vacuum stability testing was applied to a blend of samples JB2, JB5 and JB6 (Table 2). The JB2 sample was added to the blend because insufficient quantities of JB5 and JB6 were available for testing. The test was conducted on two replicate samples; hence two values are presented. In the vacuum stability test, the pressure at the beginning of the test (P_{start}) was 2 atmospheres (atm), rising to 333 atm (replicate 1) and 387 atm (replicate 2) at the end of the test (P_{end}).

The volume of gas liberated upon heating was greater than the stability limit of commercial nitrocellulose≤2 cm^3 g^{-1} (Fidanovski et al. 2016) confirming that the improvised nitrocellulose is less stable than commercial nitrocellulose manufactured using common nitrating agents. The Vacuum Stability results from a commercial nitrocellulose containing 12.6% N showed a gas evolution of 1.7 cm^3 g^{-1} suggesting that the stability of improvised NC is slightly poorer than the commercial grade nitrocellulose (Table 3). The low stability of improvised NC may be due to the presence of impurities from the drain cleaning acid used for synthesis.

Table 3. Hazard properties of improvised and commercial nitrocellulose samples. *vacuum stability tests were also performed on commercial cellulose, and the threshold is ≤2 cm^3 g^{-1} (table).

	Rotter impact test (FoI)	Mallet friction test (FoF)	Temperature of ignition test (°C)	Electrostatic discharge test (J)	Vacuum stability test (cm^3 g^{-1})
Improvised nitrocellulose	22.8 (±0.17)	>4.5	180	4.5 sensitive	2.8, 3.4
Commercial nitrocellulose	23	>4.5	170	4.5	1.7

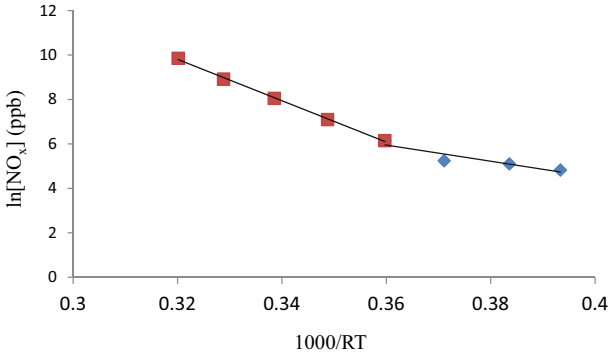


Figure 2. Arrhenius plot of NO_x evolution from nitrocellulose at different temperatures.

Chemiluminescence Analysis

The chemiluminescence of the improvised nitrocellulose sample at different temperatures was used to investigate the temperature dependence of the decomposition kinetics. The sample was heated from 0 to 100°C at intervals of 10°C and an Arrhenius plot was constructed using NO_x gas evolved (NO and NO_2) as a function of heating (Figure 2).

Within a low temperature range (0–60 °C), nitrate esters decompose via the favored hydrolytic route involving the rupture of RO – NO_2 bonds to form oxides of nitrogen (NO_2) and alkoxy radicals (RO·) followed by the oxidation of RO· to ROO· and the formation of NO (Kimura 1989).

The activation energy for the hydrolytic breakdown of the O – NO_2 bonds in the improvised nitrocellulose was~36 kJ mol^{-1} which is at the lower end of the values reported for commercial nitrocellulose containing 12.6% nitrogen (37–41 kJ mol^{-1}) (O´keeffe, Wallace, and Gill 2016). Consequently, the activation energy above 60 °C (60–100 °C) was~93 kJmol^{-1} and was attributed to the thermolytic decomposition of the O – NO_2 bonds, but this is lower than the value of 145 kJ mol^{-1} reported for commercial nitrocellulose (Bohn and Volk 1992). The lower activation energy observed for improvised nitrocellulose suggests that the decomposition of nitrate esters was due to the autocatalytic reaction of the unstable nitrocellulose. The autocatalytic reaction in improvised nitrocellulose confirms that the improvised materials will have a much shorter shelf life and will be more prone to accidental initiation than commercial nitrocellulose. Therefore, the prolonged storage of improvised nitrocellulose and any samples with unknown history is a safety concern and such materials must be handled with great care.

Conclusion

The energetic, stability and hazard properties of improvised nitrocellulose in relation to a better-characterized commercial grade of nitrocellulose with

similar nitrogen levels (12.6%) have been evaluated in order to assess the risk of these improvised materials. The improvised nitrocellulose samples were analyzed to assess their response to thermal, electrostatic, and mechanical impulses. DSC data suggested that the thermal decomposition temperature and energy content were comparable in both types of materials, and that the enthalpy of decomposition in the improvised nitrocellulose samples increases with the degree of nitration. The hazard and safety properties such as sensitivity to impact, friction, temperature of ignition and ESD suggest that improvised nitrocellulose has similar hazard characteristics to commercially available military-grade nitrocellulose. However, vacuum stability and chemiluminescence analysis revealed that improvised nitrocellulose samples are less stable than the commercial nitrocellulose, and that improvised nitrocellulose is more vulnerable and will have a shorter shelf life. The prolonged storage of such materials could therefore result in accidental initiation. This study demonstrates the need to apply extreme caution during the transport, storage, and handling of improvised nitrocellulose whether synthesized for research and development purposes or recovered from an illegal possession.

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Disclosure statement

No potential conflict of interest was reported by the authors.

Statement of Novelty

Nitrocellulose is a sensitive energetic material produced commercially from cotton linter and wood pulp, but improvised forms can be made from household chemicals. The stability, sensitivity and hazardous properties of improvised nitrocellulose are not reported in the literature, but this information is needed to ensure such materials can be handled safely when recovered by law-enforcement agencies. Our comprehensive analysis, including activation energy and vacuum stability data, provides high-impact results indicating the caution required for the transport, storage and handling of improvised nitrocellulose. This will be of particular value to readers interested in controlling and mitigating the risks of hazardous materials.

Stability and Hazard Properties of Improvised Nitrocellulose

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Research Highlights

(1) Improvised nitrocellulose samples were manufactured from household ingredients
(2) Decomposition temperatures/energies and hazard characteristics were determined
(3) Activation energies for hydrolytic and thermolytic decomposition were determined
(4) Improvised nitrocellulose was less stable than commercial-grade material
(5) We propose recommendations for safe handling of improvised nitrocellulose samples

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