



Forensic methodology for the thermochemical characterization of ANNM and ANFO homemade explosives



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ABSTRACT

Measurements were carried out to obtain thermal signatures of the most commonly used homemade explosive (HME) materials, i.e., ammonium nitrate/nitromethane and ammonium nitrate/No. 2 diesel fuel oil, using a novel laser-heating technique referred to as the laser-driven thermal reactor (LDTR). Experiments were performed for different compositions, initial masses, and steady-state temperatures, along with the effects associated with HME aging. For ammonium nitrate/nitromethane (ANNM), the NM/ANNM mass fractions investigated were 29% (stoichiometric value) and 14%; these experiments were with fresh mixtures. The 29% NM/ANNM mass fraction mixture was also aged to 3%. For ammonium nitrate/fuel oil (ANFO), a fresh stoichiometric FO/ANFO mass fraction of 6% was used initially, and aged mixtures were then investigated for mass fractions of 5.4%, 4.7%, 4.4%, and 3.7%. The results indicated that the LDTR thermograms (i.e., sample temperature change with time) for the mixed ANNM were different than the individual isolated components, and that the technique was sensitive to varying HME composition. In addition, changes in the thermograms as ANFO aged were attributed to the varying volatility of the fuel hydrocarbon fractions, and thus provide important information for forensics analysis as to the HME reactivity.

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1. Introduction

1.1. Background

Unlike standard explosive operational procedures, homemade explosives (or improvised explosives) vary widely with regard to composition, morphology, and preparation. Currently, forensic identification of homemade explosives (HME) is inadequate for determining the procedures used during their formulation, storage, and deployment [1]. Needed is a repository of data characterizing HME behavior, which can be used by the forensic science community, law enforcement organizations, and military agencies to interpret pre- and post-blast forensic evidence, assist in developing insights and protocols to disrupt/discourage HME production [2–4], and provide for safe HME disposal.

A common formulation used widely by terrorists is the mixing of ammonium nitrate (AN) with a number of different fuels to make oxidizer-fuel explosives. Ammonium nitrate is often mixed

with No. 2 diesel fuel oil (FO) or nitromethane (NM); terrorists tend to use whatever fuel is readily available [5]. Countermeasures to restrict their distribution have focused on limited AN access and fertilizer production with inert additives to inhibit AN explosive capability [2]. The explosive properties of ANFO and ANNM mixtures have been studied extensively; besides its use in illegal activities, ANFO and NM also have civilian applications (i.e., ANFO for blasting, mining, and mineral extraction applications [6,7], and NM for fuel in motor racing).

The ANFO detonation behavior is considered by theoreticians to be non-ideal because detonation reaction kinetics are relatively slow (long detonation zone) and dependent on a variety of physical parameters [8,9]. Such explosives are generally high-porosity, low-density materials for which the fuel and oxidizer are not mixed on a molecular level [10]. They exhibit low detonation velocities and larger detonation reaction zones. A review of ANFO is presented by Hurley [11], which describes various physical parameters (e.g., charge diameter, fuel oil content, particle size, particle size distribution, loading density, moisture content, porosity, additives, and charge confinement) that influence detonation properties. Prill morphological effects (i.e., bulk density, surface features, shape) on explosive behavior were investigated by Rao et al. [12]. They argue that the maximum energy release will depend on stoichiometric fuel/oxygen mixing and maximizing the surface area throughout

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Nomenclature

a, b	Coefficients
A	Sample geometric cross-sectional area [m^2]
$c_p(T)$	Specific heat capacity [$\text{J g}^{-1} \text{K}^{-1}$]
(dT/dt)	Sample temperature-time derivatives [K s^{-1}]
$F(T, T_0)$	Heat transfer term [W]
I_l	Intensity of the laser beam that heats the sample [W m^{-2}]
k_c	Coverage factor
$m(t)$	Sample total initial mass [g]
n	Number of samples
$q(T)$	Specific heat release rate due to chemical reaction [W g^{-1}]
Q	Total specific heat release (or absorption) [kJ g^{-1}]
R_{lh}	Laser heating rate (defined by the baseline thermogram maximum slope) [K s^{-1}]
s	Standard deviation
t	Time [s]
t_1, t_2	Time corresponding to the sample temperatures T_1 and T_2 , respectively
T	Temperature [K]
T_0	Sample temperature of the baseline [K]
T_r	Reactor surface temperature [K]
T_1, T_2	Sample temperatures at two different laser fluences [K]
u_c	Combined uncertainty
w_i	Mass fraction ($=m_i/m_{\text{total}}$ where i is a chemical component of the total composition)

Greek symbols

$\beta(T)$	Absorptivity
λ	Laser wavelength [m]
ΔH	Change in enthalpy [kJ g^{-1}]
Δm	Mass of the reactive portion of the sample, mass loss [g]
ΔT_i	Change in temperature between values $i = 1, 2$ [K]
$\Delta (dT/dt)_i$	Change in the temperature-time derivative between values $i = 1, 2$ [K s^{-1}]
$\tau(T)$	Temperature-dependent relaxation time [s]

Subscripts

AN	Ammonium nitrate
ANFO	Ammonium nitrate/diesel fuel oil mixture
i	Initial
f	Final
FO	Diesel fuel oil
NM	Nitromethane
sas	Sample and substrate
so	Substrate only

the prill. Thompson and Shan [13] have observed that the interface region between the AN and FO has the highest temperatures and chemical exothermic reactivity. Miyake et al. [14] used different tube materials to study the effect of confinement on ANFO detonation. They found that the confinement of ANFO had a strong influence on the detonation velocity (a measure of the quantity of energy released [15]) and the detonation front curvature. Bohanek et al. [7] investigated the influence of the initiation energy on ANFO detonation velocity and found that a minimum quantity of explosive charge is required to initiate an explosion of ANFO when confined to a steel pipe. Ammonium nitrate characteristics (such as grain size, density, porosity) have been investigated regarding their effect on ANFO detonation velocity. For example,

Zygmunt and Buczkowski [4] found that the ANFO detonation velocity increases significantly with increased AN granule porosity and decreased size, and that the ANFO stoichiometric fuel/oxidizer ratio had the highest detonation velocity regardless of AN porosity. Cooper et al. [16] used a velocity interferometry system to investigate the effect of prill compaction and prill-to-prill interactions on detonation by low-stress impact. They observed post-shock particle velocity variations and in- between-prill jetting. Dobrilović et al. [15] investigated the effect of the charge temperature on detonation velocity. For ANFO confined within a steel pipe, the results indicated a linear increase in detonation velocity with temperature. Jackson et al. [10] and Janesheski et al. [17] investigated the effect of confinement-tube wall thickness on detonation velocity. Both studies concluded that the average detonation velocity increased and the wave front curvature decreased with increasing tube thickness. The thicker tubes better confine the energy losses to the axial direction (limiting radial losses).

Trace detection of explosives is difficult, with nearly every analytical chemical method having been employed to this problem [18,19]. As discussed above, the non-ideal behavior of ANFO, and other similar HMEs, make it difficult to predict their thermochemical and thermo-physical characteristics (especially for ANFO which has been investigated extensively in the past, e.g., [20,21] over a variety of relevant physical conditions [1]). To address this issue, a data measurement approach is needed that 1) operates over physical conditions and heating rates relevant to HME applications, and 2) provides a means for comparative analysis of existing HME mixture formulations.

1.2. Investigation objectives

In an earlier investigation [22], HME precursors were studied individually, i.e., ammonium nitrate (AN) and nitromethane (NM), using a novel laser-based technique (referred to as the laser-driven thermal reactor) to characterize material thermochemical/thermo-physical behavior. The technique provides temporally resolved temperature-time thermograms, which are used to identify trends and characteristics unique to each HME chemical mixture. The objective of the current study was to evaluate the thermal decomposition of HME oxidizer-fuel mixtures, i.e., ammonium nitrate/nitromethane (ANNM, a single composition fuel) and AN mixed with No. 2 diesel fuel oil (ANFO, a multi-hydrocarbon composition fuel) with respect to sample steady-state temperature, initial mass, and fuel/mixture mass fraction. Also, discussed are issues associated with sample aging and changes in the mixture composition and volatility with time.

2. Experimental arrangement

2.1. Brief facility description

The LDTR experimental facility and theory of analysis are detailed in Nazarian and Presser [22,23]. Some pertinent information is summarized for clarity, along with a description of the current facility modifications for these experiments. The laser-driven thermal reactor consists of a sphere-shaped, gold-plated, copper-foil reactor (with an outer diameter of 18.2 ± 0.1 mm and thickness of 0.14 mm) mounted within a vacuum chamber, along with optical, gas supply, and computer-controlled data acquisition subsystems (see Figs. 1 and 2). The reactor is open on the top and bottom for access to within the sphere. The sample is supported on a customized K-type fine-wire thermocouple (0.25 mm bead diameter, unshathed, and 0.200 s \pm 0.002 s response time) stationed at the center of the reactor (defining the 'sample' temperature). A second thermocouple is

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