



Detection of explosives – Studies on thermal decomposition patterns of energetic materials by means of chemical and physical sensors



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ABSTRACT

The present study investigates the feasibility of library free discrimination between energetic and non-energetic materials, based on the behavior of the substance rather than its chemical composition. This allows the evaluation of explosion hazard even for novel, previously not encountered materials. The setup is designed for solid samples in the lowest milligram range and utilizes rapid heating for thermal activation of the analyte. The reaction is monitored by a pressure sensor, photodiodes and metal oxide semiconductor (MOX) gas sensors. Key features are the application of readily available physical and chemical sensors, utilization of ambient air as a carrier gas, regulation of sample weight by the geometry of the heater and easily understandable read out. The acquired data from physical sensors is analyzed for specific thresholds to separate high reactive energetic materials and propellants. The remaining analytes are investigated with MOX gas sensors in the second step to discriminate less reactive energetic materials from benign ones. The rate of correct assignments for the developed setup is 93,2% with a total of 11 false negatives and 2 false positives out of 190 experiments. For high reactive energetic materials principal component analysis (PCA) shows potential for the identification of the analytes.

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1. Introduction

The encounter of unknown items in public areas like airports and railway stations has often raised security concerns. Even though the most items have proven to be harmless afterwards, the risk of encountering an improvised explosive device (IED) still exists. A quick and reliable threat assessment of unknown substances from suspicious items regarding their explosion hazard is a difficult challenge. To deal with this challenge, a new experimental approach has been taken to distinguish between hazardous energetic materials and benign materials. Nowadays numerous methods are used to detect bulk explosives including but not limited to application of gas sensors [1,2], Raman and infrared spectroscopy [3–5], ion mobility spectroscopy [6–8], liquid or gas chromatography cou-

pled mass spectrometry [9,10] and explosive-sniffing dogs [11,12]. Except the use of canines, all methods mentioned above (including the one developed in this work) imply sampling and the investigating the sample rather than the whole loading. Most of them are reliable but still have some weak points such as low time efficiency and the usage of libraries. In this work the authors especially address the last point, as nowadays it is crucial to be able to detect unknown and previously untested explosives.

Based on the previous work by Maurer et al. [13] physical and semiconductor metal oxide (MOX) gas sensors are combined within a single setup to build up a system which is capable to distinguish between hazardous energetic materials and benign ones. Initial experiments performed by Maurer dealt with the question, whether rapid detection and characterization of small quantities of solid powdery explosives (about 1 mg) is possible by measuring the changes in pressure and the emission of visible or infrared radiation while heating the samples. The objective was to develop a laboratory system with the ability to detect and classify explosives irrespective of the presence of certain molecular groups by characterizing the performance of the material if stimulated through rapid heating (more than 3000 K/s). It was shown that reliable discrimination only by means of physical sensors is not possible as the detection rates for some secondary explosives are very low. Due to the large variety of explosives and their properties, a reliable

Abbreviations: MOX, metal oxide semi-conductor gas sensor; PCA, principle component analysis; IED, improvised explosive device; HMTD, hexamethylene triperoxide diamine; PETN, pentaerythritol tetranitrate; TATP, triacetone triperoxide; Amm, Ammongelit; Han, Hanal[®]; RDX, cyclotrimethylene-trinitramine; HMX, cyclotetramethylene-tetranitramine; BP, black powder; Tetryl, trinitrophenylmethyl nitramine; SGP, smokeless gun powder; TNT, trinitrotoluene; DNT, dinitrotoluene; Sem, Semtex 1a; AN, ammonium nitrate.

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Table 1
Gas sensors, used for the investigation of the gas phase.

| Name | Produced by | Sensitive to | U(heater) [V] |
|---------|-----------------|-------------------------------|---------------|
| ASMLK | Applied Sensors | CH ₄ | 2,3 |
| UST7333 | UST | NO _x | 2,3 |
| UST3430 | UST | C _n H _m | 3,5 |
| Fig2620 | Figaro | CO | 5,0 |

detection based on only one or two detection methods is difficult. The addition of MOX gas sensors that analyze the gaseous reaction products of the thermal decomposition to complement physical sensors can overcome this problem. In the work of Maurer et al. [13] MOX gas sensors were used in a separate experiment.

After redesign of the system by combining physical and MOX gas sensors in one single setup a significant raise of detection rates has been achieved and thus the feasibility to distinguish between energetic and benign substances has been proven. In addition, the constructed system shows potential for classification or even identification of explosive materials.

2. Materials and methods

In this section the experimental setup, sensors of choice and the applied heater are presented. Also the process of producing and collecting data is described and the investigated analytes are listed.

2.1. Experimental setup

The central parts of the hardware are the explosion chamber and the gas sensor chamber which are connected via a valve. The detailed scheme of this setup is presented in Fig. 1.

The explosion chamber (3) is equipped with a heater and three physical sensors (IR-diode, pressure sensor, VIS-diode) and is attached to two 2-way solenoid valves (2a, 2b) which are used to set the path of the air flow either through this chamber for sample withdrawal and gas phase analysis (red route) or through a bypass, allowing the reaction to occur in a sealed environment and regenerating the MOX gas sensors (blue route). Latter are placed in the gas sensor chamber (4). An air sample pump (model SKC 3755G, flow rate 2 l/min.) (5) is attached for transportation of the ambient air (carrier gas) through the test setup – there is a continuous carrier gas flow both during and between measurements. The entering ambient air is filtered by a washing bottle filled with activated charcoal (1).

2.1.1. Sensors' characteristics

For measuring the emissions, IR and VIS photodiodes were used to cover the whole range of wavelengths between 200 and 1750 nm. One diode was a silicon detector with UV enhanced response (200–1100 nm, sensitivity of 10 V/μA); the other – an InGaAs diode (800–1750 nm, sensitivity of 10 V/μA). Both diodes were manufactured by Edmund Optics, USA. Pressure was measured by a gauge pressure sensor produced by Honeywell, USA (type 26PCBFA6G, max. pressure 5 psi/34.47 kPa, sensitivity of 0.01 mV/Pa). The MOX gas sensors used for the investigation of the gas phase are listed in Table 1. All gas sensors were operated at the heating voltage proposed by manufacturer.

2.1.2. Heater characteristics

For heating samples micro-structured hotplates produced by CAESAR (Center of Advanced European Studies and Research, Germany) were used. A cavity measuring 1 × 1 × 0.4 mm was etched into a SiO₂-substrate for holding the test agent. A Nickel heating structure was placed on the bottom side of the hotplate. The detailed description of the heating system was given by Mau-

rer et al. [13]. The heaters were used as a disposal, utilizing a new one for each experiment.

2.2. Program sequence and data acquisition

Each test series of a substance consisted of ten experiments. Each experiment consisted of a preliminary measurement (determination of the explosion chamber background) followed by the measurement of a corresponding analyte. At the end of each test series a flushing run was performed (regeneration of the gas sensors, cleaning the system). The electrical resistance of the gas sensors was continuously measured during the experiment but also during the flushing run in order to see if the reaction chamber and the pipes were sufficiently clean at the end of the process. The sampling rate of the resistance measurements for all MOX gas sensors was 2 Hz. The heater was exchanged after each experiment. In the first step of each preliminary measurement both solenoid valves were set to bypass for 33 s. (see Fig. 1, valves 2a and 2b, blue path). Subsequently the valves were switched to explosion chamber for 30 s (red path in Fig. 1) and finally back to bypass for another 30 s. The heater was not turned on during the preliminary measurements. After that the measurement of an analyte started. The valves were set to bypass for 60 s after which the heater was turned on for 1 s at 45 V. Simultaneously data acquisition from IR/pressure/VIS sensors started and continued for 3 s at sampling rate of 1 kHz. In the next step the gas flow was directed to the reaction chamber for 300 s for sample withdrawal to detect any gaseous reaction products, if present. In the last step the valves were switched to bypass for 900 s allowing the MOX gas sensors to regenerate. Introduction of the next sample to the system and the exchange of the heater were also carried out in this step. At the end of each test series a flushing run was performed.

The measurements and control over the experiment were performed using the LabView software from National Instruments. For data acquisition, the PC interface cards PCIe 6251 and PCI 6122 were used.

2.3. Sample preparation

The indentation in the middle of the heater was used for sample volume restriction, which indirectly limited the maximum weight. The dent was always filled completely resulting in sample weight in the range between 150 and 400 μg. The reason for this approach is the consideration that in an operational area an exact measurement of the sample weight would be very difficult and should be avoided. The samples were not homogenized in order to simulate the operational area conditions.

2.4. Test agents

A total of 18 substances were investigated in course of this work. 13 were energetic and 5 benign materials. This resulted in a total of 180 experiments. Additionally blank measurements (no analyte) were performed. The used materials are listed in Table 2.

All explosives except TATP and AN were provided by the German Armed Forces. TATP was synthesized according to Matyas and Pachmann [14] and AN was readily available in the laboratory. The selection was based on following criteria: for explosives it was intended to cover the most common representatives categorized by different sensitivity for ignition; for benign materials it was decided to focus on those ones which are optically similar to homemade explosives and can be easily used in IED mock-ups.

2.5. Principle component analysis (PCA)

PCA was carried out using XLSTAT software (version 2013.4.07).

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