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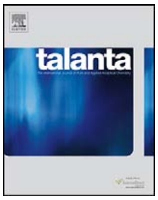

Ultra trace detection of explosives in air: Development of a portable fluorescent detector

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ABSTRACT

This paper describes a system for the detection of nitroaromatic explosives consisting of a portable detector based on a specific fluorescent material. The developed sensor was able to perform an ultra trace detection of explosives, such as trinitrotoluene (TNT) or its derivate 2,4-dinitrotoluene (DNT), in ambient air or on objects tainted with explosives. In the presence of nitroaromatic vapors, the fluorescence of the material was found to decrease due to the adsorption of nitroaromatic molecules on its specific adsorption sites. The sensor exhibited a large sensitivity to TNT or DNT at their vapor pressures (respectively 6 and 148 ppbv) and the detection threshold was evaluated on a laboratory test setup and was found to be 0.75 ppbv for TNT. Moreover, the detector demonstrated no loss of performance in the presence of humidity or interfering compounds. All the tests led to the conclusion that the sensor fulfills the main requirements for the identification of suspect luggage, forensic analyses or battlefields clearing.

1. Introduction

During the past 10 years, counterterrorism activities have been strengthened. An increasing use of explosives by terrorists and the need to eliminate unexploded landmines has rendered the detection of such substances crucial in order to ensure civilian security. Consequently, one strategy of law enforcement is to develop reliable, sensitive and specific systems for the detection of explosives in luggage, vehicles and soils. There already exist various valid technologies for the analysis of explosives: HPLC, EC, GC/MS, IR, XR imaging, NMR and IMS [1]. However the cost, the portability and the complexity of many of these methods are unfit for on-site monitoring devices.

As a result, much interest has been focused on the development of portable, low-cost and reliable detection devices. A suitable sensor would also be expected to analyze samples in a relatively short time. In the particular case of landmine detection, a system that has no physical contact with the target is required [2]. A wide variety of chemical sensors have been developed based on various transductions. Mass-sensors have suitable detection properties but in most cases they fail to detect explosives with very low vapor pressures. Optical devices based on immunosensors exhibit high selectivities thanks to the specific reaction of antibodies with explosives molecules [3,4]. The ELISA device (enzyme-linked immunosorbent assays) exhibits a detection sensitivity of 1–15 ng/mL of TNT, in 5 h [5]. This technique is dedicated to the detection of explosives in a liquid matrix. Despite these advances, there is currently a strong demand for explosives vapor sensors that are more suitable for the inspection of landmine detection, forensic analysis and suspicious luggage. Previous studies have demonstrated the potential of optical sensors for such detection [6–8].

TNT represents a significant target when carrying out detection on bombs hidden in soil or luggage. This is due to TNT being one of the most commonly used explosives for military applications; a great part of the landmines currently in use contains trinitrotoluene [9]. TNT is also well known for its toxicity with regard to human health and the environment [10]. A secondary product of TNT synthesis plays an important role in the detection of TNT vapor: 2,4-dinitrotoluene (DNT). As the vapor pressure of this substance is two orders of magnitude higher than that of TNT (i.e., 148 as opposed to 6 ppbv at 20 °C [11]), DNT has a significant part in the chemical signature of TNT [12–14].

In this context, the present study describes the development of an optical device that combines all the expected properties, and that is based on the capability of π-conjugated fluorescent materials to interact with explosive vapors, in particular nitroaromatic compounds (NACs) [15,16]. In the presence of NACs, π-conjugated fluorescent materials have exhibited excellent fluorescence extinc-

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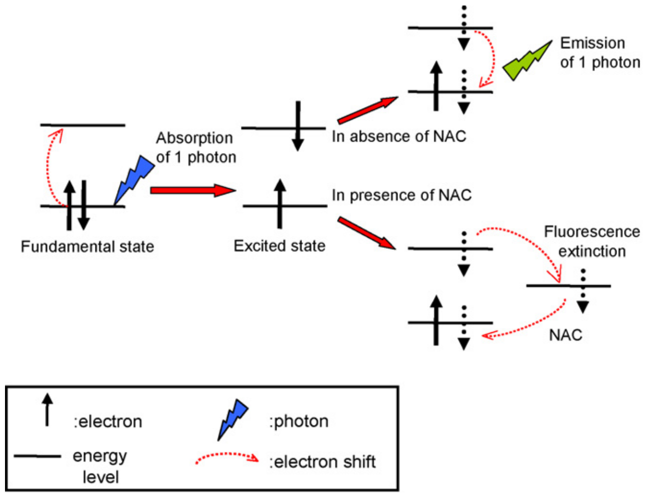


Fig. 1. Fluorescence and the charge transfer principle.

tion properties due to a charge transfer mechanism [17,18], as reported in Fig. 1.

2. Experimental

2.1. Material description

The selected material was a π-conjugated phenylene–ethynylene diimine and its chemical structure is given in Chart 1. The elaboration of this compound was performed according to details described elsewhere [19]. The fluorescence properties of the selected material were brought on by the π-conjugated phenylene–ethynylene part, while the spatial structure was a result of the cyclohexyl functions. The high electron density of the diimine provided it with a strong affinity towards electron deficient compounds such as nitroaromatics. The adsorption of NAC molecules on the diimine surface involved a decrease in the fluorescence intensity (Fig. 1).

The spectroscopic film properties, i.e., absorption and emission, were determined respectively on a Perkin–Elmer Lambda 35 and on a Jobin-Yvon Fluoromax 3. The material was deposited on the entire surface of a glass substrate (Heathrow Scientific microscope slides 75 mm × 25 mm × 1 mm) by spin-coating (Braive Instrument's spin-coater @ 600 rpm) from a 2.5 mg/mL THF solution during 60 s of drying. The fluorescence quantum yields of the films were recorded on a Hamamatsu C9920-02.

2.2. Prototype description

The transduction and detection techniques have been previously developed for a fluorescence-based array biosensor [20], and were here adapted for a single chemical gas sensor. A schematic of the setup is depicted in Scheme 1.

The central element was a conventional microscope slide (Heathrow Scientific, 75 mm × 25 mm × 1 mm) that acted both as the transducer and the substrate of the active material. A typical device was composed of four sensing areas of 1.4 mm^2, and each of these sensing areas was successively orthogonally excited using focused led emission. Finally, the detection of the corresponding fluorescent emission was synchronized.

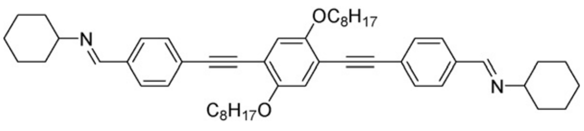
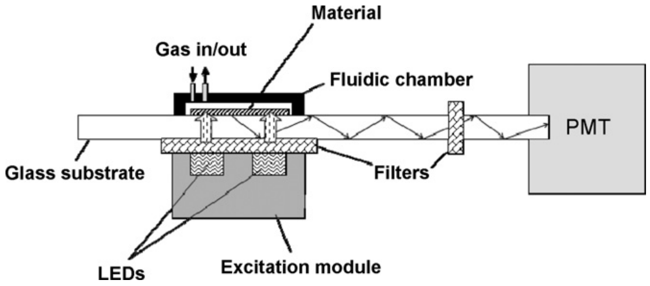


Chart 1. The chemical structure of the diimine.



Scheme 1. A schematic representation of the prototype instrumentation. Fluorescence excited by focused and filtered light emitting diodes (LEDs) was guided by total internal reflection in the glass substrate up to a photomultiplier. ⇨ : light from the LEDs; ↗ : light from the material (fluorescence).

In order to clarify the results, the decision was made to represent a single area signal. The excitation module (Scheme 1) was composed of four low-cost light emitting diodes (NSHU550A, NICHIA Corporation) delivering an optical power of 0.6 mW at a wavelength of 378 nm. Light was focused on the sensor areas through two ball lenses (uncoated, 5-mm diameter, LaSFN9, Melles Griot) with a spot diameter of 1.47 mm (full-width at half maximum). A single band pass filter (330WB80, Omega Optical) was incorporated between the two series of lenses to reject wavelengths below 250 nm and above 400 nm. The illumination time was 22 ms, and the delay between two acquisitions was 60 s.

Fluorescence was guided in the slide by total internal reflection and was directly collected at the end-face by a photo-multiplier tube (H8249-101, Hamamatsu). The detector had been interfaced with a bandpass filter (400ALP/E, Omega Optical) and a high pass colored filter (OG570, Schott) to suppress the remaining excitation radiation. The high collection efficiency of the transducer rendered the use of imaging optics unnecessary, thus increasing the robustness, compactness, and simplicity of the system. The transducer was embedded into a home-made fluidic reaction chamber fabricated in aluminum and coated with black PTFE. The flow channel was 6 mm in width, 0.130 mm in height, and 46 mm in length. The sample successively passed the areas #1, #2, #3, and #4. The fluidic circuitry was composed of 1/8″ PTFE tubes, and the whole prototype was 40 cm × 30 cm × 16 cm (a photography of the prototype is available in Supporting Information: Fig. S1).

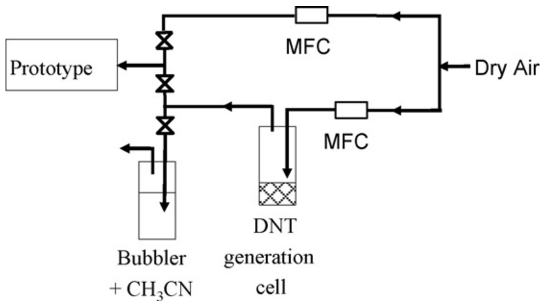
The system was controlled with a single-board computer (BL2120 Smartcat, Z-World, USA) that had been programmed using Dynamic C® language. This board had a LCD/keypad window where the measurements were displayed. Another computer was also interfaced using an RS232 serial bus, and Microsoft® Office Excel macros were written to interact with the single-board computer. The developed software was able to display real-time graphics and to select experimental parameters such as PMT settings and the measurement frequency.

2.3. Test descriptions

Caution: TNT is highly explosive and must be handled with care.

With the exception of TNT, which was homemade for one batch and military supplied for the other, all chemicals were purchased from Aldrich Chemical Co. Inc. and used as received.

Diimine was evaluated for the detection of DNT under dry synthetic air and real ambient air. The detection of TNT was only investigated under real air at ambient conditions. For the tests under real ambient air, peristaltic pumps were used to generate the air flows. The rate of humidity was close to 50% (±10%) and the



Scheme 2. The detection setup used for the tests under dry air.

temperature was 24 °C (±2 °C), as determined by means of a probe TESTOSTOR 171-2.

The general testing procedure was:

- 30 min of stabilization under air (synthetic or ambient)
- 10 min of exposure to explosives
- At least 20 min under air (synthetic or ambient)

The sensor response was then expressed as a percentage of the fluorescence inhibition after 10 min.

2.3.1. System qualification

2.3.1.1. Qualification in the laboratory. The setup described in Scheme 2 was used to generate nitroaromatic vapors under dry air conditions (Air Liquide Synthetic Air, Alphagaz Air 1). The air passed through the generation cell containing 1 g of DNT with a flow rate of 20 L/h. By means of mass flow controllers (Bronkhorst EL-FLOW mass flow controller (MFC) and E7100 Flow Bus) and valves, the generated vapor could either be distributed to the prototype (sensor experiments) or bubbled in a reactor full-up with HPLC-grade acetonitrile (cf. concentration measurements).

For the generation of nitroaromatic vapors in a real atmosphere, the prototype and the DNT bubbler were concurrently equilibrated by means of peristaltic pumps (Masterflex 7524-40) with a flow rate of 20 L/h. During the exposure, the prototype line was connected to the generation cell (see Scheme S1, Supporting Information).

2.3.1.2. Concentration measurements. The generated DNT vapor was calibrated by first carrying out a 1.5-h sampling in a bubbler containing 20 mL of acetonitrile (Aldrich, HPLC grade). This was followed by analyses on a UV spectrometer (Perkin–Elmer Lambda 35). Five standards were prepared with a concentration of 0.5, 1, 1.5, 2 and 4 mg/L of DNT in acetonitrile. The maximum absorbance was observed at 244 nm.

2.3.1.3. Qualification on real explosives and selectivity evaluation. In order to qualify the system under real ambient air, tests were carried out on a number of analytes placed in 20 mL vials. The sampling was achieved by placing the gas inlet above the opened vials (see scheme S2, Supporting Information). The tested analytes included DNT, TNT (two batches), dimethyldinitrobutane (DMNB), methylene chloride, methylethylketone (MEK), toluene, Scorpio® perfume, Lacoste® perfume, laundry detergent, sun screen, and then DNT again coming from the same batch (Table 1).

2.3.2. System improvement

The air sampler was improved by adding a cone-shaped apparatus to the gas inlet. Several stainless steel cones, differing in their dimensions, were used. These apparatuses are indexed in Table 2.

The performances of each apparatus were determined by carrying out sampling above a 54 mm glass Petri dish containing 100 mg of DNT (see Scheme S3, Supporting Information).

Table 1
Values of vapor pressure of tested analytes at 20 °C.

Compound	Vapor pressure (ppbv); [Ref]
DNT	148; [11]
TNT	6; [11]
Dimethyldinitrobutane (DMNB)	2700; [21]
Methylene chloride	$4.6\ 10^8$; [22]
Methylethylketone (MEK)	$0.9\ 10^8$; [22]
Toluene	$0.3\ 10^8$; [22]
Scorpio® perfume	Unknown
Lacoste® perfume	Unknown
Laundry detergent	Unknown
Sun screen	Unknown

2.3.3. System performances

Detection tests were carried out on clothes tainted with NAC (DNT and TNT). During the sampling, the cloth was placed under the gas inlet equipped with the best cone-shaped apparatus (see Scheme S4, Supporting Information). Clothes tainted with explosives were prepared in one of two ways:

(1) NAC, in solid state, was deposited on a clean piece of cotton cloth (100% cotton N°OF 0608CM00288, 4 cm × 4 cm) and then removed by shaking the cloth. This way, only impregnated particles remained. Following this operation, approximately 5–10 mg of NAC was left on the cloth.
(2) A clean piece of cotton cloth was soaked in a solution of DNT or TNT in HPLC-grade acetonitrile (5 mL, 10 mg/mL) and then dried. The remaining nitroaromatics were estimated to be 50 mg.

3. Results and discussion

3.1. Fluorescence properties of diimine

Diimine absorption and emission spectra are available as Supporting Information: Fig. S2. The material presented four main absorption bands in the solid state: 332, 383, 394 and 415 nm. The optical densities of the films ($\lambda = 415$ nm) were above 0.2 which corresponded to a thickness between 50 and 100 nm [19]. The diimine exhibited a greenish-blue fluorescence with a maximum at 480 nm and the photo luminescence quantum yield of the material was measured to 40% while deposited as a thin film on a microscope slide.

According to the excitation and emission filters described previously, the spectral properties of the material made it suitable for its use in the prototype.

3.1.1. System qualification

3.1.1.1. Qualification in laboratory. The evolution of the intensity of fluorescence of ten diimine coatings under dry air is reported in Fig. 2.

When exposed to dry air, the signal was found to be very stable; no decrease of the fluorescence was recorded. Under the conditions of the measurement (illumination time: 22 ms, period: 60 s), the photobleaching of the thin film was negligible. When exposed to 120 ppbv of DNT, the fluorescence was detected to decrease

Table 2
Sizes of the cone-shaped apparatuses.

Entry	Diameter (cm)	Height (cm)
1	4	10
2	8	10
3	4	5
4	2	10
5	2	5

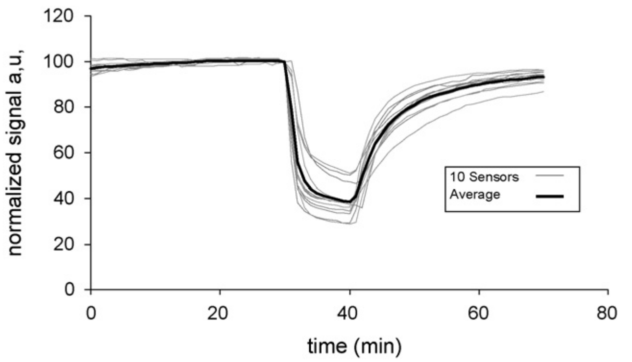


Fig. 2. The evaluation of the performances of 10 sensors and the average signal.

slowly and after 10 min, the average sensor response was 61% ($\sigma = 7$).

The whole process, including the elaboration of the sensitive film, the fluorescence measurement and the gas generation was thus well reproducible and suitable for further industrial developments.

As expected, fluorescence quenching was observed with DNT. The diimine film was found to be very sensitive. Concerning the reversibility, it was quite slow—nevertheless, after 30 min, the system had recovered 90% of its original signal, indicating that the sensor was suitable for multiple detections. Strong interactions were involved in the detection process, e.g. π–π interactions between the rich electron ring of the diimine and the poor electron ring of the DNT [23].

The evolution of the fluorescence of the coating under dry air or real ambient air of a diimine thin film is plotted in Fig. 3.

The behavior of the fluorescence under real air was close to that observed under dry air. The only difference concerned the value of the fluorescence inhibition: 54% for dry conditions as opposed to 41% for real conditions. This slight decrease was attributed to a competitive adsorption of other vapors, such as volatile organic compounds (VOCs) contained in the atmosphere and hygrometry, onto the diimine surface. Nevertheless, the large signal observed under real air demonstrated that the adsorption of water was obstructed by the hydrophobicity of the material. This compound was thus suitable for the detection of DNT in very polluted and complex gaseous environments.

3.1.1.2. Concentration measurements. The calibration curve of DNT in acetonitrile is available as Supporting Information: Fig. S3.

The solution obtained after bubbling in acetonitrile contained only DNT at a concentration of 1.63 mg/L. This corresponds to 120 ppbv in air at 22 °C—a value very close to that reported by Pella [11], i.e., 148 ppbv.

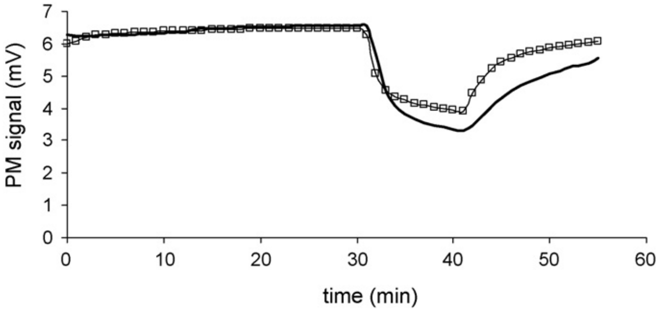


Fig. 3. The comparison of performances between dry and ambient air. ━━ : testing under dry synthetic air; —□— : testing under ambient air.

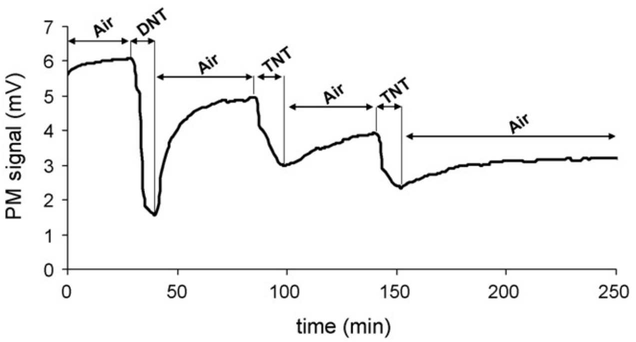


Fig. 4. System qualification on real explosives under real conditions.

3.1.1.3. Qualification on real explosives and selectivity evaluation. The evolutions of the intensity of fluorescence under various vapors under real ambient air are plotted in Figs. 4 and 5 (the whole experiment is represented in Fig. S4, Supporting Information).

Explosive vapors. The fluorescence was found to slowly decrease when exposed to DNT and an equivalent behavior was recorded under TNT. Diimine was thus sensitive to various nitroaromatic explosives. The response to TNT was 40% whereas it was 82% for DNT. The weaker effect of TNT was attributed to the lower vapor pressure of TNT as compared to DNT (6 ppbv as opposed to 148 ppbv at 20 °C [11]). The reversibility of the adsorption of TNT vapors was much slower than that for DNT vapors: after 1 h, the recovery was still not complete (almost 48%). This specific reactivity was due to a better sensitivity of the diimine film towards TNT than towards DNT. Because the film was electron rich, it had stronger affinity with TNT. With the presence of his three nitro groups, TNT central aromatic ring is more electron deficient than the one of DNT, this resulted on a stronger TNT adsorption and so in a worse desorption. Thus, the material is more sensitive to TNT than to DNT. Nevertheless, the second exposition to TNT gave rise to a similar response (38%). The solid film had not become saturated with TNT after one exposition.

If one considers the results obtained for TNT and DNT at their vapor pressures (82% for DNT (148 ppbv) and 39% for TNT (6 ppbv)), as well as the noise level of the system (1.5%), the detection threshold for a signal/noise ratio of 3 could be estimated to 9 ppbv for DNT and 0.75 ppbv for TNT.

Volatile organic compounds and domestic volatile compounds. No significant effect was observed when the fluorescent film was exposed to high concentrations of DMNB, toluene, CH_2Cl_2 or the other domestic volatile compounds. Concerning the test with MEK vapors, the signal was unstable and irreproducible. This effect might have been caused by the process of vapor generation (see Section 2.3.1.3). It was not particularly suitable for the well-defined generation of vapors with high vapor pressures such as MEK

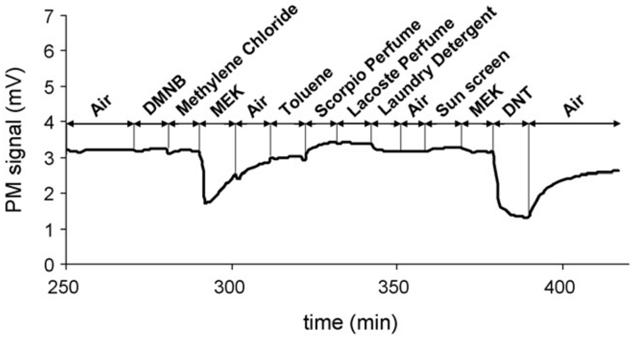


Fig. 5. System qualification for several volatile compounds under real conditions.

Table 3
The performances of the cone-shaped apparatuses.

Entry	Cones dimensions		Sensor response (%)
	Diameter (cm)	Height (cm)	
0	Without cone-shaped apparatus		24
1	4	10	34
2	8	10	10
3	4	5	38
4	2	10	15
5	2	5	21

(93,000 ppmv at 20 °C). The irreproducible fluctuation of the intensity of fluorescence under MEK could be attributed to a strong variation of the pressure in the measurement cell. Nevertheless, no stable or major quenching was observed when the material was exposed to any of these organic vapors. The selectivity of the sensor was thus excellent with regard to complex atmospheres such as perfumes.

A last test with DNT was performed; the fluorescence inhibition was here 56% (to be compared to 82% for the first measurement). This result suggested that the sensor remained very sensitive after being contaminated with large amounts of VOCs. The decrease in the sensor response was caused by irreversible adsorptions of certain VOCs (or previous exposures to NACs), which obstructed the adsorption of DNT.

Two important features were the results of these experiments:

- The sensor was sensitive enough to detect TNT in a complex, polluted and contaminated atmosphere.
- The sensor selectivity was sufficient enough to reduce the amount of false alarms in complex and fluctuating environments.

3.1.2. System improvement

Previous investigations have been devoted to ways of increasing the detection sensibility by means of improved sampling systems [24,25]. For this purpose, cone-shaped samplers were tested in order to improve the sensitivity of the detection device. These samplers were of varying heights and diameters, and a large range of shapes and volumes could thus be obtained. The sizes of each cone and their corresponding detection results with respect to DNT in a glass Petri dish are reported in Table 3 (detection tests curves are available as Supporting Information: Fig. S5).

All tests demonstrated a fluorescence inhibition, with more or less intensity, depending on the cone shape. The improvement in sensor response was only achieved in two cases, i.e., with cones 1 and 3. Among all the tested cones, cone 3 gave the best results. At a constant diameter, the response decreased with an increasing height. The fluorescence inhibition was reported to be a function of D^2/h, where D is the cone diameter and h is the cone height.

The response did not seem to be correlated to the cone volume, whereas the correlation between the fluorescence inhibition and D^2/h exhibited an optimum. From Fig. 6, it appeared that the optimum cone had a value of D^2/h equal to 3.24. The correlation between D^2/h and the response of the sensor was not clear and work is currently ongoing to link this geometrical parameter to a fluidic parameter of the air collection. Nevertheless, the cone with $D = 4$ cm; $h = 5$ cm was used for the further detection tests.

3.1.3. System's performances

The fluorescence inhibition of the coating when exposed to polluted clothes is listed in Table 4 (the detection curves are presented in Fig. S6, Supporting Information). Previous tests showed that no effect was observed when the detector was exposed to the single cotton cloth or to the cotton cloth polluted with acetonitrile.

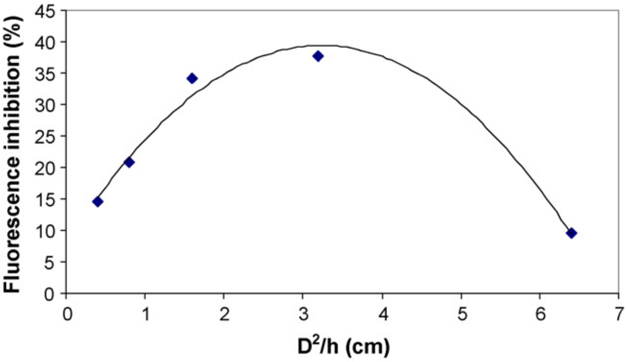


Fig. 6. The fluorescence inhibition (%) as a function of the geometrical factor D^2/h (cm).

Table 4
Results from the detection on a piece of tainted cloth.

Entry	Analyte	Deposition method	Fluorescence inhibition (%)
0	DNT	Powder (result from Table 3)	38
1	DNT	Solid	32
2	TNT		14
3	DNT	Solution	48
4	TNT		17

The signal was found to significantly decrease in the presence of DNT- or TNT-tainted cloths. The detector was thus deemed to be sensitive enough to detect devices or materials polluted with trace amounts of explosives, and should thereby be suitable for the detection of suspect luggage or for the clearing of battlefields.

The obtained responses depended on the NAC compound as well as on the techniques of preparation. DNT vapors gave rise to the largest values (32–48%), whereas those obtained for TNT vapors were much lower (14–17%). Here again (see Section 3.1.1.3), this feature was attributed to the lower vapor pressure of TNT as compared to DNT. The reversibility was also incomplete in the case of TNT.

The test on 100 mg of explosive powder gave rise to a larger response as compared to that on the cotton polluted with solid (5–10 mg). Furthermore, concerning the techniques of preparation, solid deposition led to the weaker responses (e.g. DNT: 32%). The best signals were obtained with the solution impregnation (e.g., DNT: 48%), while the response of the powder was intermediate (e.g., DNT: 38%). The amount of vapor collected by the cone apparatus depended on the amount of solid explosive as well as on its evaporation. The evaporation process was assumed to be controlled by the NAC dispersion.

In the case of the cotton tainted with a solution, the particles of the explosives that came from the acetonitrile solution were embedded in the cloth. It was thus believed that this gave rise to a better evaporation as compared to the NAC powder. This effect was certainly prevailing and led to the best response.

4. Conclusion

The present paper dealt with the study of an explosives vapor detector based on a fluorescent material: a diimine phenylene–ethynylene compound. The thin film was deposited on a glass substrate that acted as a wave-guide. This portable detector was dedicated to the identification of suspect luggage or to mine clearance in battlefields. The air collection of the detector was optimized, and with a well-adapted cone-shaped apparatus, the miniaturized detector exhibited a very large sensitivity and selectivity towards nitroaromatic explosives (TNT or DNT):

- When exposed to DNT in a real and polluted atmosphere, the quenching of the fluorescence was found to be 38% for 10 min.
- The sensor was sensitive enough to detect trace amounts of TNT on a piece of cotton.
- No response was recorded when the sensors were exposed to large concentrations of domestic vapors such as perfumes or solvents.
- The detector matched the main requirements for the identification of suspect luggage, the clearing of battlefields or forensic analyses.

Future investigations will concern the material and the prototype:

- The ageing of the material will be studied. The ageing of phenylenevinylene compounds is well documented [26,27], whereas no study deals with the ageing of our type of fluorescent materials: phenyleneethynylene.
- The prototype will be integrated in a single device, equipped with a pump, a battery and an alarm based on suitable data processing. The evaluation of the temperature and humidity rate on the behavior of the prototype will be investigated.
- In this paper we focused on the use of one material and the detection of one kind of explosives, but the sensor is composed of four sensing areas. In further studies, we will investigate other fluorescent materials in order to be able to detect more than just one kind of explosive. After the development of a suitable deposition process (i.e. ink-jet) we will be able to develop a multi-sensor based on 4 fluorescent materials to detect and also identify various explosives.
- New materials will be developed to detect other compounds, such as toxics VOCs.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.talanta.2009.12.040.

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