

Standoff Detection and Identification of Explosives and Hazardous Chemicals in Simulated Real Field Scenario using Time Gated Raman Spectroscopy

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ABSTRACT

The detection of hazardous chemicals, explosives, improvised explosive materials, energetic materials and their associated compounds for security screening, forensic applications and detection of unexploded ordnance is an active area of research. The results based on comprehensive experimental study and performance of time gated Raman spectroscopy (TGRS) for stand-off detection of explosives and hazardous chemicals under realistic scenario are presented. Representative results drawn from the experimental study for detection of explosives and hazardous chemicals in simulated real field scenario are given.

Keywords: Explosives detection; Hazardous chemicals; Time gated Raman spectroscopy; Standoff detection; Real field scenario

1. INTRODUCTION

The rising terrorist activities in worldwide have urged for the development of modern and easily transportable fully automated detection systems for rapid and reliable standoff detection and identification of almost all kind of explosives and harmful chemicals in real field scenarios¹⁻⁸. In some scenarios like airports, metro stations, railway stations, markets and shopping malls, remote explosives detection techniques can be used in which the person investigating suspicious materials maintains a safe distance from the object being screened. In remote detection techniques the detection unit may approach the suspicious object closely. While in some other important real field scenarios like terrorist and maoist effected areas and forests, inspection may be required from true standoff distance. If the explosives detection system is fully automated and vehicle mounted then the operator can detect and identify the suspicious materials by operating the detection system from their position.

Accurate and rapid identification of explosives and hazardous chemicals outside the typical laboratory environment and under potentially dangerous conditions is a major technological challenge. Standoff detection of explosives in real field scenarios requires the development of transportable, fully automated, compact and reliable detection systems, based on modern analytical techniques. A suitable real time detection unit should be able to distinguish these harmful materials from safe or innocuous substances with a reasonable level of confidence. Detection of almost all kind of explosives

and hazardous chemicals from safe standoff distance in real field scenarios becomes extremely challenging because of the variety of variable explosives, improvised explosive materials and many of unknown background materials that may have interfering Raman and fluorescence spectral features⁹⁻¹².

Many advanced standoff atomic and molecular spectroscopic techniques such as time gated laser induced breakdown spectroscopy (TG-LIBS), laser photoacoustic spectroscopy (LPAS) and time gated Raman spectroscopy (TGRS) can be used to detect and identify suspicious materials from safe standoff distance. Atomic and ionic emission spectral lines from laser induced plasma in TG-LIBS provides elemental information of the samples. This technique does not provide the direct information about explosive molecule. Sensitive LPAS technique is based on vibrational absorption and it requires the tunable QCLs in mid IR region. Most of the glass and plastic material do not have transmission in mid IR region. Detection of explosives and hazardous materials through transparent and translucent glass bottle is not possible using this technique.

Molecular spectroscopic Raman scattering technique is virtually instantaneous, non-destructive in nature and provides very high selectivity. It provides the unique fingerprint of the of explosives and other chemicals without any need of tunable laser. Because of its capability to provide unique set of vibrational frequencies (fingerprint) to identify the molecular species¹³, backscattered Raman spectroscopy has become an important method for defence and forensic applications¹⁴⁻¹⁵. Molecular spectroscopic techniques like spatially-offset Raman spectroscopy (SORS), Nano-particle based surface enhanced Raman spectroscopy (SERS) and time gated Raman

spectroscopy (TGRS) are different advanced variants of conventional backscattered Raman spectroscopy. SORS is very promising technique to identify explosive materials through translucent plastic containers from short distance. SERS is very highly sensitive technique for trace detection of explosives and other chemicals that relies on the enhanced Raman scattering of molecules that are adsorbed on SERS active surface, such as nanostructured silver or gold. TGRS is one of the most suitable laser-based analytical technique for real time standoff detection and identification of suspicious materials. It generally involves gated and intensified CCD as a detector and nanosecond pulsed laser as an excitation source. Using automated standoff TGRS technique, suspicious sample in different scenarios can be investigated in real time from safe distance without putting the individual and vital properties in the danger zone.

2. TIME GATED RAMAN SPECTROSCOPY (THEORETICAL BACKGROUND)

For detection of explosives and hazardous chemicals from close distance, light weight and hand-held detection systems based on conventional back-scattered Raman spectroscopy can be made using a compact CCD coupled spectrograph and a compact continuous wave (CW) laser. This type of hand-held explosives detection unit can be made quite portable. The major problem in using conventional Raman back-scattering is its very low signal strength relative to high fluorescence noise of the background material or the sample itself that in many important applications may screen the weak Raman signal of the targeted sample. Using a compact CW laser as an excitation source and a compact CCD coupled spectrometer as a sensor, weak backscattered Raman scattering of the sample is collected along with strong fluorescence (if present) noise of background or targeted material.

In many important standoff applications measured weak backscattered Raman signal of suspicious material is generally masked by the strong fluorescence noise of background material or targeted materials and huge ambient light. Fortunately, laser induced fluorescence and Raman process have different lifetime. Raman scattering process is instantaneous, whereas laser induced fluorescence emission is relatively slow process¹⁶. Inelastically backscattered scattered Raman photons are observed instantly during excitation of the sample, whereas strong laser induced fluorescence signal (if present) can still be observed after several nanoseconds, or even milliseconds in many cases. Thus, if we irradiate the suspicious material with a nanosecond laser pulse, the inelastically backscattered scattered Raman photons from the sample will be generated during the laser pulse only, whereas most of the laser induced fluorescence photons will be emitted for long time after the nanosecond laser pulse. This long-lived fluorescence signal can be suppressed using proper nanosecond-time window in intensified CCD detector. Time gating of intensified CCD detector can be synchronised with the nanosecond laser pulse to restrict spectroscopic data collection to the very small-time window where inelastically backscattered Raman photons from the targeted standoff suspicious sample are likely to reach the gated detector, while excluding the huge ambient noise and strong laser induced fluorescence light outside of this time window.

Correct synchronisation between the excitation laser pulse and receiving time window or gate width in gated and intensified CCD allows effective filtration (rejection) of the possible long-lived fluorescence signal coming from the background materials as most of this fluorescence photons reach the gated detector after the backscattered Raman photons. The graphical representation of effect of time gating process is as shown in Fig. 1. In addition, the strong ambient light can be rejected between two consecutive laser pulses using proper time delay and narrow collection time window or gate width in gated and intensified CCD (ICCD) detector. After rejecting the huge ambient light and strong laser induced fluorescence content of the background, the collected backscattered Raman signal of the suspicious sample can be intensified using high gain in the intensifier tube of ICCD.

A trolley mounted and transportable detection system based on TGRS technique was developed and tested in simulated real field scenarios for detection and identification of little amounts of explosives and their derivatives on relevant surfaces at a distance of 10 meter. This simulated real field detection scenario was developed in laboratory.

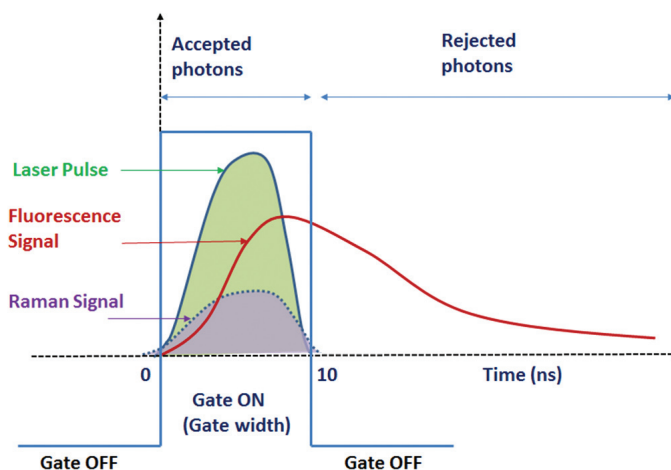


Figure 1. Time gating principle of ICCD.

3. STANDOFF DETECTION SYSTEM DESCRIPTION

In this section, we shall describe the design technique of TGRS prototype for standoff detection of explosive and hazardous materials in real field scenarios. A schematic diagram of TGRS standoff prototype unit using nanosecond pulsed laser and spectrograph equipped with gated and intensified CCD (ICCD) detector is as given in Fig. 2.

This detection unit consists of a nanosecond pulsed Nd:YAG laser operating at second harmonic, 8-inch reflected type receiving telescope and gated ICCD coupled Czerny turner spectrograph. A frequency-doubled Nd:YAG pulsed laser (wavelength: 532 nm, pulse duration FWHM: 4 ns and maximum repetition rate: 10 Hz) was used as a excitation source to produce stand-off Raman spectra of samples. Raman backscattered signals from the chemical samples were collected using 8-inch aperture reflected type motorised receiving telescope arranged in a co-axial geometry. The co-axial geometry allows variable standoff spectroscopic

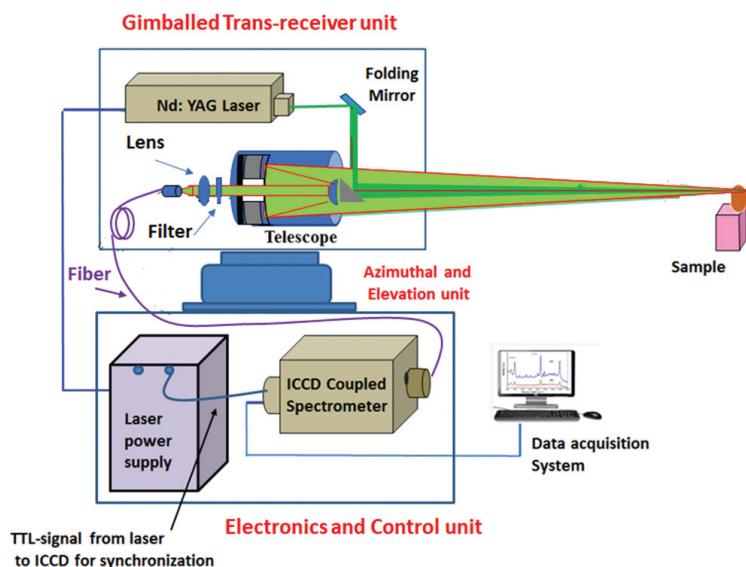


Figure 2. Schematic of standoff detection system.

measurement from the suspicious samples at different distances without re-alignment of the optical sub-system. A convex lens focuses the collected backscattered Raman photons and Rayleigh scattered photons along with laser induced fluorescence as well as ambient light from the output of the receiving telescope to the bundle of optical fiber. A long pass notch filter at 532 nm wavelength was inserted between the output port of the receiving telescope and focusing convex lens to block strong Rayleigh scattered photons. An optical fiber bundle of length around 2 m was used to transport the collected light from the exit port of the telescope to the entrance slit (slit size :20 μm) of gated ICCD coupled spectrograph. Grating having 300 lines/mm and blazed in visible region was used as a dispersive element in the spectrograph. Stand-off Raman spectra of the samples were recorded using a thermoelectrically cooled and gated intensified CCD. The stand-off and time gated detection was carried out by triggering the gated and intensified CCD (ICCD) camera using Q-switched output TTL pulse from the laser. Depending on standoff distance of the suspicious materials and the laser pulse duration, suitable time delay ($t = 2d/c$, where d is the distance of target and c is the velocity of light) and gate width (of the order of laser pulse duration) were used in gated ICCD. To reject the ambient light and long tail of laser induced fluorescence signal, gate delay of approximately 66 ns and gate width of 10 ns (comparable with laser pulse duration) were used in ICCD for 10-meter distance. All time-gated standoff backscattered Raman experiments in simulated real field scenarios were carried out in day light condition.

Standoff TGRS experiments were performed on p-nitro benzoic acid (PNBA) and Ammonium Nitrate (AN) in pure form and deposited or scattered on different background materials (grass, wood, road, soil, plastic etc.), from a standoff range of 10 meters. In these experiments 100 millijoule pulse energy was used to induce Raman effect. Receiving telescope and excitation laser were mounted on a single platform. This platform has manual

azimuthal and elevation capability. By using elevation and azimuthal feature of the prototype, the laser pulse was directed towards suspicious sample. Samples were irradiated using nanosecond laser pulse and back reflected Raman signals were collected using telescope arranged in co-axial geometry as shown in Fig. 2. Laser spot size (diameter) at 10 m was around 15 mm.

To adjust the focal point, the inter mirror separation of the telescopic mirror can be adjusted depending upon the sample position from the detection unit for proper collection of the weak back scattered Raman signal with minimum ambient noise.

4. RESULTS AND DISCUSSION

Raman spectra of PNBA and AN in pure form and deposited on different background surfaces were recorded from a distance of 10 m. Figures 3 and 4 depicts the recorded spectra of PNBA in pure form and deposited on different background surfaces.

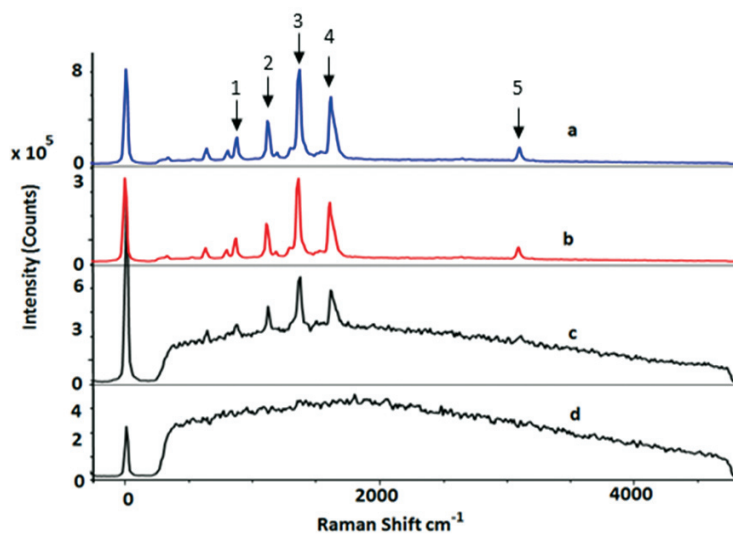


Figure 3. Recorded Raman spectra of (a) PNBA, (b) PNBA on glass slide, (c) PNBA on road surface, and (d) Road surface.

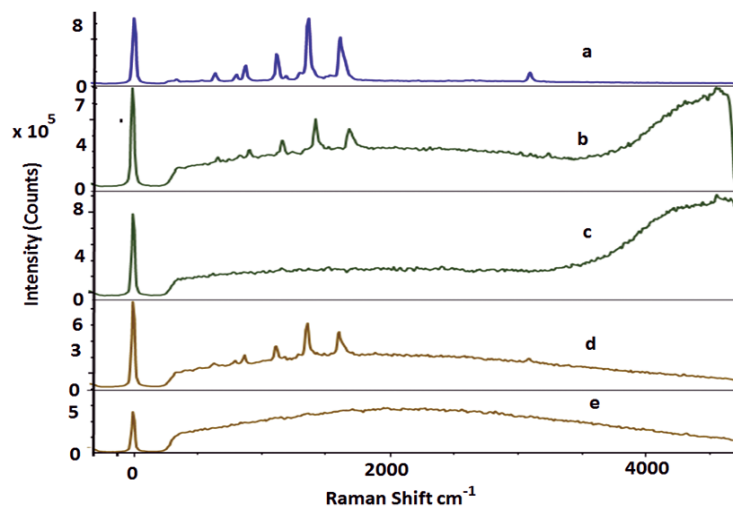


Figure 4. Recorded Raman spectra of (a) PNBA, (b) PNBA on grass, (c) grass, (d) PNBA on wood surface, and (e) Wood surface.

Figure 5 depicts the recorded spectra of AN in pure form and AN deposited on different background surfaces. To record the Raman spectra of pure samples, 5 gm quantity of PNBA and AN were used. Accumulations of Raman signals for 10 pulses were used to record standoff Raman spectra of pure samples. To record the standoff Raman spectra of samples deposited on different background surfaced 50 mg quantity of AN and PNBA were deposited on 20 mm x 20 mm area of background substrate. Density of deposited materials were around 12.5 mg/cm². For each measurement of deposited materials 100 pulses were irradiated on target samples and backscattered Raman signals were accumulated. Recorded standoff Raman spectra of PNBA and AN were analysed using available data of nitroaromatic compounds and inorganic nitrate salts¹⁷⁻²⁵. Main Raman peaks of PNBA and AN with peak position and assignments are listed in Table 1. All Raman peaks of PNBA also appeared in case of PNBA deposited on glass slides, road, grass and wood as shown in Figs. 3 and 4. All Raman peaks of AN also appeared in case of AN deposited-on plastic but only peak Nos 1 and 2 appeared in case of AN mixed with soil as shown in Fig. 5. It is because of the low intensity of Raman lines at these positions and small amount of AN mixed with soil. Road, wood, grass and soil give fluorescence signal as shown in Figs. 3 and 4 but plastic material gives Raman signature along with fluorescence as shown in Fig. 5. PNBA and AN deposited on different background give detectable Raman signature as shown in Figs. 3, 4, and 5.

The peak assignment corresponding to the most intense peak in Raman spectra of AN and PNBA are as summarised in Table 1.

LabVIEW based data acquisition and analysis software was developed and used to acquire and analysis the Raman spectra and to identify the suspicious samples. Figure 6 shows the representation of GUI of the detection system software.

Raman spectra of several explosives, hazardous and non-hazardous materials were recorded to build the Raman data library for real time standoff identification of the explosives and hazardous chemicals. This software performs the Raman data analysis using advanced peak finding technique to identify the explosive and hazardous materials in real time. Standoff backscattered Raman spectra of several explosives, improvised explosive materials and hazardous chemicals were recorded on different background surfaces in simulated real field conditions. This TGRS system is capable to identify the suspicious samples from standoff distance depending on the strength of Raman spectra of sample as well as the Raman or fluorescence content of background material.

Table 1. Assignment of most intense peak of Raman spectra of AN and PNBA

Molecule	Peak No.	Raman band position (strength)	Assignment
PNBA	1	865 cm ⁻¹ (moderate)	COO ⁻ bending vibration
	2	1113 cm ⁻¹ (strong)	C-H in plane bending/ring breathing
	3	1255 cm ⁻¹ (strong)	Symmetric NO ₂ stretching vibration
	4	1608 cm ⁻¹ (strong)	C-C stretching of Benzene ring
	5	3085 cm ⁻¹ (moderate)	CH vibration
AN	1	714 cm ⁻¹ (weak)	Plane bending of nitrate ion (NO ₃ ⁻)
	2	1045 cm ⁻¹ (strong)	Symmetric NO ₃ ⁻ stretching vibration
	3	1416 cm ⁻¹ (weak)	Asymmetric NO ₃ ⁻ stretching
	4	1459 cm ⁻¹ (weak)	NO ₄ ⁺ (ammonium ion)

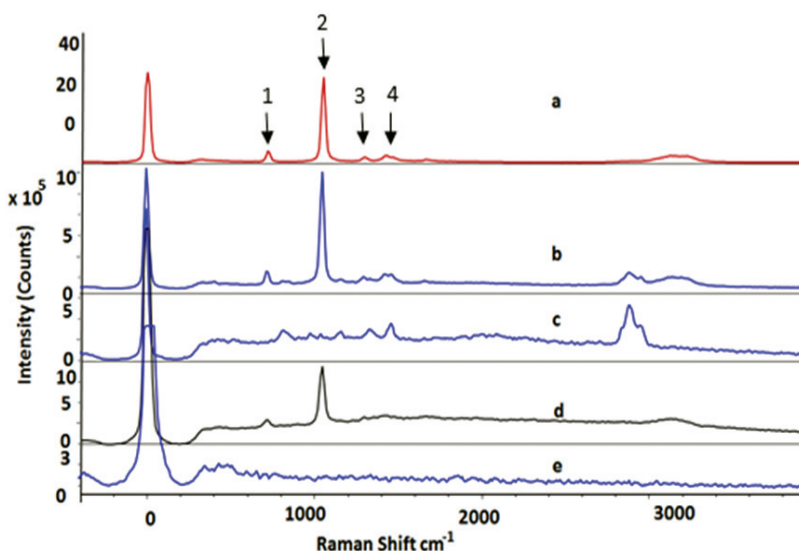


Figure 5. Recorded Raman spectra of (a) AN, (b) AN on Polypropylene (PP) plastic, (c) Polypropylene (PP) plastic, (d) AN on soil, and (e) Soil.

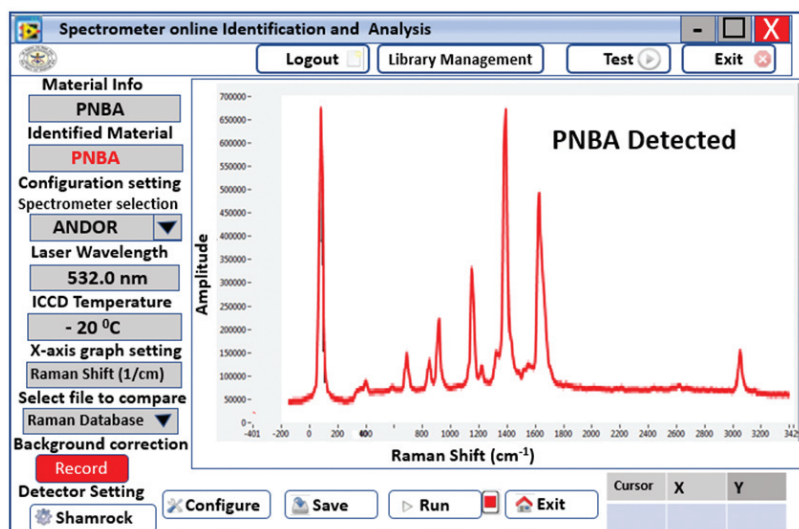


Figure 6. LabVIEW based GUI showing the detection of PNBA.

5. CONCLUSIONS

Using time gated Raman spectroscopy (TGRS) based system several explosives, improvised explosive materials and hazardous chemicals can be detected in pure form as well as materials scattered or deposited on different background surfaces depending on the strength of the Raman signal of sample as well as content of Raman / fluorescence strength of the background materials. This technique has excellent potential for in-situ standoff detection of explosives / chemicals deposited on different background surfaces from standoff distances. System-based on TGRS can be used to capture the weak Raman spectra buried in high background noise. Detection system-based on TGRS can be developed for in-situ real time detection of explosives and hazardous chemicals. For defence and security applications, trolley mounted, tripod mounted, and vehicle mounted trace explosives detection system can be developed based on time gated Raman spectroscopy.

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