



Trace detection of explosive and their derivatives in stand-off mode using time gated Raman spectroscopy



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ABSTRACT

Trace detection is utmost important for detection of concealed explosive materials in form of IEDs. Surfaces of suspicious objects may be scanned for contamination of explosives. Sensitivity and selectivity are two main concerns in field of explosive detection. Raman spectroscopy provides remarkable selectivity. Time-gated Raman spectroscopy also has potential of providing sensitivity for trace detection. In present study, time-gated Raman spectroscopy investigated for detection of ppm level concentration of explosives. To achieve better sensitivity, various parameters of the experimental set-up like gain of intensifier, number of pulse accumulated and pulse energy of laser were optimised. The trend of change in intensity of Raman scattering (background subtracted) was observed by varying values of these above mentioned parameters. Followed by optimization of these parameters for better sensitivity, Raman spectra of RDX samples at lower concentrations (prepared by mixing RDX with KBr) were recorded. The corrected intensity increased in linear fashion with increase in gain of intensifier, number of pulses accumulated and pulse energy. After that, Raman spectra of RDX were recorded from a stand-off distance of 5 m in backscattered mode with concentrations down to 100 ppm.

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1. Introduction

Since last two decades, terrorism has evolved as the most challenging menace against humanity. Most of the terrorist attacks have been noticed for use of explosives materials stored in some concealed form like metal container etc. Such types of bombs are known as improvised explosive devices (IEDs). Till today the methods used require approaching the suspicious object in order to perform the analysis with a great risk to the operator. Therefore, a new technique, need to be developed to detect and identify target object with acceptable level of confidence maintaining safe distance for the operator [1–4].

Standoff explosive detection involves methods for sensing the presence of explosive devices when vital assets and those individuals monitoring, operating, and responding to the means of detection are physically separated from the explosive device. The zone of severe damage varies with scenario and bomb type, it may vary from 10 m for a pedestrian suicide bomber and 100 m for a vehicle-based bomb [5]. Standoff detection and identification of

such explosive materials is one of the most unavoidable but greatest challenging tasks for researchers and engineers.

The detection at large distances involves physical difficulties like i. the intensity of the return light decreases inversely with the distance squared and ii. absorption & scattering losses in air lead to significant compromise in sensitivity.

Laser-based spectroscopic techniques such as laser-induced breakdown spectroscopy (LIBS), Raman spectroscopy (RS), resonant Raman spectroscopy (RRS), photo-dissociated/laser-induced fluorescence (PD/LIF) and laser-based photo-acoustic spectroscopy (LPAS) have the potential to detect minute amount of explosive materials on contaminated surfaces left by handling etc. [6–9].

LIBS technique involves power density of the order of GW/cm² to generate plasma of the target sample [10–12]. This level of power density is achievable with short, high peak power laser pulses and tight focusing of the laser beam by transmitting optics (usually spot size of the order of few microns). LIBS signal comprises elemental information i.e. spectral lines in LIBS spectra gives information of elements presents in the test sample whereas explosive detection requires molecular information [13,14]. It is destructive technique and hence not suitable to examine people and vehicles. Since laser beam is tightly focused into a small spot, it is difficult and time-taking to scan large objects in real conditions

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[15]. Photo-dissociation Laser-induced fluorescence (PD-LIF) involves decomposition (dissociation) of polyatomic molecules into characteristic fragments [16–18]. PF-LIF method can be used to detect only nitrogen-containing explosives. In addition, the dissociation of different molecules may require different level of energy and wavelengths. In addition, absorption characteristics of species generated in dissociation process of different molecules shall be different. Thus, PD-LIF requires a tunable laser source. In addition, it involves excitation with UV wavelength and hence thermal degradation of the sample may occur [19,20]. Laser-photo acoustic spectroscopy (LPAS) involves the vibrational absorption and hence requires the tunable laser source in mid IR range i.e. 5–12 μm which corresponds to 830–2000 cm^{-1} . Quantum-cascade lasers are the main contestant for LPAS due to their compactness and light weight [21–25]. The main concerns with QCLs are their requirement of cooled environment and limited tenability [26]. In addition, detection of suspicious materials through even transparent glass and plastic containers is not possible. The sensitivity is adversely affected by humidity.

Raman spectroscopy is one of the effective techniques for detection and identification of explosive materials [27–30]. This technique is suitable for detecting both organic and inorganic explosive materials in form of solid, liquid and powder. A single-wavelength laser is sufficient to generate Raman scattering of all the molecules of interest. Raman spectroscopy is a molecular spectroscopy that provides direct information about the molecular structure and composition of the material. The potential of this technique lies in its ability to locate and identify unequivocally many different compounds at a time. The process is non-destructive, sensitive, fast, and repeatable. The vibrational spectrum provided by Raman spectroscopy is a unique molecular ‘fingerprint’, that is capable of differentiating very similarly structured molecules from one another with great level of specificity in real-scenario [31–34]. Conventional Raman spectroscopy in back-scattered mode, resonance-Raman spectroscopy (RRS) [35,36], surface-enhanced Raman spectroscopy (SERS) [37–39], spatially-offset Raman spectroscopy (SORS) [40–42], time-gated Raman spectroscopy [21,27,29] are being explored for detection of explosives. Time-gated Raman spectroscopy is the most suitable technique for stand-off detection of explosive materials. It involves pulse laser source and intensifier-charge coupled device (ICCD). ICCD is being synchronized with optical laser pulse with appropriate delay depending upon mainly stand-off distance [21,36,43].

In stand-off Raman spectroscopy, a pulsed laser source of nanosecond or shorter pulse is preferred as excitation source as it provides high peak power (few MW). In addition, pulse laser enables gated-detection which in turn ensures the rejection of ambient light between consecutive laser pulses and collection of Raman photons (photons generated in inelastic scattering) only during laser pulse. In this way, high power pulse with gated detection results in better signal-to-noise ratio (SNR). Raman photons are collected with a receiving optics, and then coupled to dispersive spectrograph directly or using optical fiber. Intensified CCD coupled with spectrograph is used to detect and produce Raman spectrum of the sample under study [28,44,45]. Dry sand samples having explosive concentrations 4–8% have been investigated using 532 nm as excitation laser wavelength [29]. Visibly excited stand-off detection may face fluorescence of samples and impurity. However, using pulse laser as excitation source and gated detection can be utilized to reduce contribution fluorescence and ambient light [9]. Random Raman lasing using 532 nm as excitation is being reported for stand-off explosive detection. The occurrence of Random Raman laser depends on beam-quality which is difficult to maintain at large stand-off distances [46].

Raman spectroscopy promises better sensitivity with visible wavelength as excitation compared to that in near infrared range as the Raman scattering cross-section depends inversely on the fourth power of the wavelength [31]. So far, stand-off UV Raman systems reported in the literature have been applied mainly to detection of explosives and chemicals (chemical and biological contaminants) [27,47–51]. Though, UV range excitation theoretically offers further better sensitivity, high energy UV photons exhibit potential risk of sample degradation and burning [31,52–54]. UV excitation in absorption bands of explosives causes photolysis which decreases concentration of the explosive molecules (51). Explosives exhibit problem of sample burning remarkably due to their absorption in deep UV range [55,56]. Photo-degradation of sample with UV excitation may be reduced remarkably by following two methods: (a) either sample should be in motion like open-stream flow or magnetic stirring [47] so that point of interaction of laser with samples may be varied during investigating target material (not possible in real scenario) or (b) use of pulse energy of level of micro-joule (not sufficient for stand-off detection). UV excitation based methods do not promise the detection of explosives through normal transparent glass and plastic containers. Though UV excitation promises higher maximum permissible energy (MEP) for eye safety, it may generate other irreversible biological effects in operator body [57,58]. Hence, UV excitation is not practical in stand-off where high pulse energy has to be transmitted.

Despite of several advancements in Raman spectroscopy has been achieved in the area of explosive detection, however Raman spectroscopy in the visible range remained to be investigated for trace detection of explosives. Trace detection targets surfaces of suspicious objects where there is probability of contamination like door handle of vehicles, handles of brief-case etc. The level of contamination may vary from few ng/cm^2 to $\mu\text{g}/\text{cm}^2$ (i.e. ppm to ppb level) depending on the way of handling the explosives in making IED etc and adhesive nature of explosives. In addition, the effect of various parameters of the set-up of time-gated Raman spectroscopy on the sensitivity remains to be explored. Here, the effect of various parameters like gain of intensifier of intensified charge-coupled device (ICCD), number of pulses used in accumulation and pulse energy of laser on sensitivity was investigated thoroughly. After optimization of these parameters in terms of sensitivity, RDX was detected at lower concentrations. Stand-off detection of 1,3,5-trinitroperhydro-1,3,5-triazine (RDX) up to 100 ppm level using visible Raman spectroscopy i.e. 532 nm as excitation laser beam was demonstrated.

2. Experimentation

An experimental set-up used in the present study was developed according to the schematic depicted in Fig. 1. This prototype mainly involves a pulse laser excitation source, ICCD-coupled spectrograph and a compact receiving optics. The pulse laser was a 2nd harmonic of Flash lamp-pumped Nd:YAG laser (Make: Quantel, France, model: Brilliant). It has maximum pulse energy of about 160 mJ at 532 nm, pulse duration of the order of 6 ns and beam divergence of about 1 mrad. It has variable pulse repetition rate of 1–10 Hz. In these measurements, pulse repetition rate of 10 Hz was used.

The collection optics was very compact having diameter of 1 inch and consisted of collimating optics (C), long pass filter (LPF) and finally focusing optics (F). The revived backscattered Raman signal was collected and collimated with collimating optics (C). Long pass filter (LPF) at 532 nm with very steep transition width $\sim 225 \text{ cm}^{-1}$ and high optical density (O.D.) of the order of 6 (Make: Semrock, USA) was used to reject Rayleigh scattered photons. Finally focusing optics (F) was used to focus the remaining signal

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