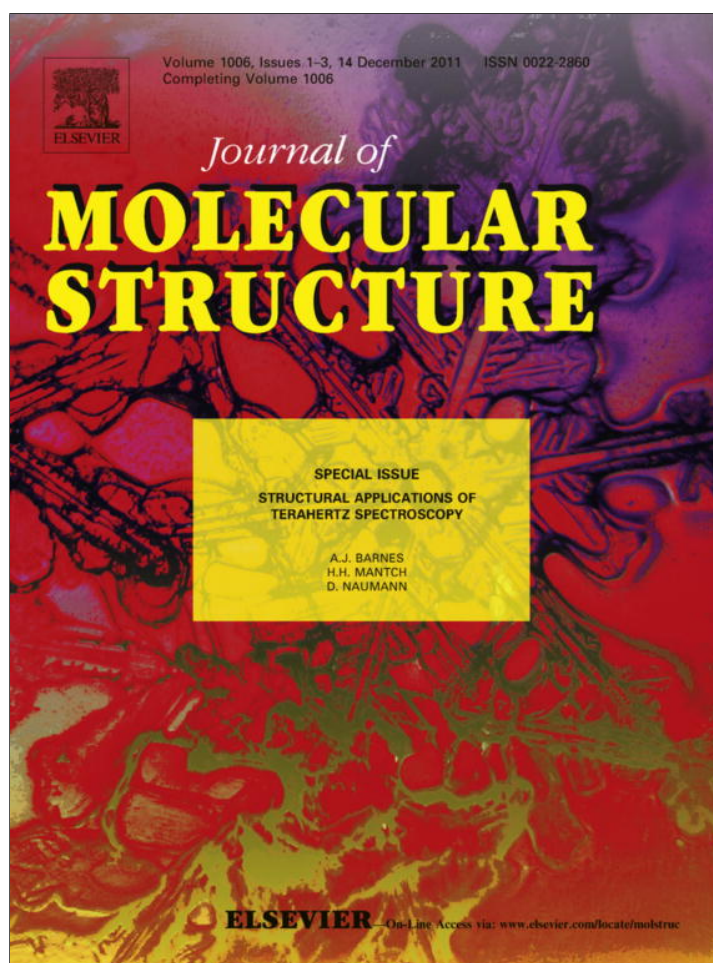




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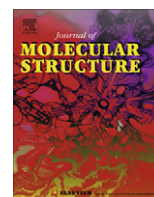
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Dendrimer based terahertz time-domain spectroscopy and applications in molecular characterization

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ABSTRACT

Electro-optic Dendrimer is used to generate milliwatts of terahertz power by difference frequency method. A terahertz time-domain spectrometer (THz-TDS) has been designed around this source that exhibits wide broadband terahertz range, ~0.1 to 35 THz. Examples of molecular characterization are discussed for three common explosives and the vibrational states of Fullerenes. The explosives' spectra are unique for each explosive that allow detection and identification of the species. The Fullerenes C₆₀ and H₂@C₆₀ also exhibit distinctively different spectra and absorbance states indicating that the THz-TDS is suitable for probing increased number of vibrational states expected from molecular vibrations.

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1. Introduction

The electro-optic (EO) route for terahertz (THz) generation is advantageous, because, in this case the pump-THz conversion is not limited either by emission saturation or by heat dissipation. In EO route, the main mechanisms include: EO rectification (EOR), difference frequency generation (DFG), and optical parametric oscillation. Of these, EOR depends on a femto-second pulsed laser (femto-laser) where an ultra-fast laser pulse is introduced into the lattice of an electro-optically active material; the lattice acts as a rectifier to convert the very high frequency derived from the femto-laser pump to a relatively lower frequency pulse that falls in the terahertz range. This is the so-called electro-optic rectification effect. The rectification method usually uses a femto-laser at 800 nm wavelength while other wavelengths, e.g., 1064 nm may also be used. The difficulty here is that, not only the process is depended on the availability of a femto-laser, but two vital parameters of the terahertz radiation – the output power and the terahertz range – are completely dependent on the characteristics of the femto-laser. As such, only low average power has been produced and a range of a few terahertz has been possible. The output is always pulsed because of the pulsed nature of the femto-laser pulse train. Because of its low available power and low THz range (~5 THz) it is difficult to uniquely characterize many materials system. A source capable of providing higher power and wider THz range in a compact form, therefore, is important for spectroscopy applications.

2. Difference frequency generation

In contrast, difference frequency generation (DFG) (or two-photon excitation) not only eliminates the use of an expensive femto-laser, it also allows for producing both continuous wave (CW) and pulsed terahertz radiation, as well as higher output power and tunable terahertz range. Early efforts have shown that DFG can be achieved by means of commercially available diode lasers. Nobel Laureate Robert F. Curl Jr. (Chemistry, 1996) group [1] has reported generation of tunable far-IR radiation by means of two single-mode diode lasers by focusing the overlapped beam in AgGaS₂ crystal. Their scheme involved two diode-lasers with collimated output, spatially overlapped and focused into AgGaS₂ crystal. The combined pump generated 3.3 nW of far-IR radiation. Shi et al. [2] have reported terahertz generation by DFG method in GaSe crystal. These authors generated an average terahertz power of 0.43 μW corresponding to an average pump power of 895 mW. Our earlier work [3] showed that chromophore doped and poled poly(amido amine) Dendrimer can produce ~3.4 mW terahertz power when pumped by two fiber coupled diode lasers with a combined pump power of ~5.5 W. Thus the terahertz figure of merit ($P_{\text{THz}}/P_{\text{pump}}^2$) of this source is $1.124 \times 10^{-4} \text{ W}^{-1}$. This was achieved by means of the higher electro-optic coefficient of the EO Dendrimer, viz., 130 pm/V [3]. While 3.4 mW average THz power is a significant improvement over EOR or photoconduction, another important factor for terahertz applications, such as time-domain spectrometry, is the obtainable range from a given source. Both EOR and photoconductor sources span only up to a few terahertz. However, many molecular systems become indiscernible

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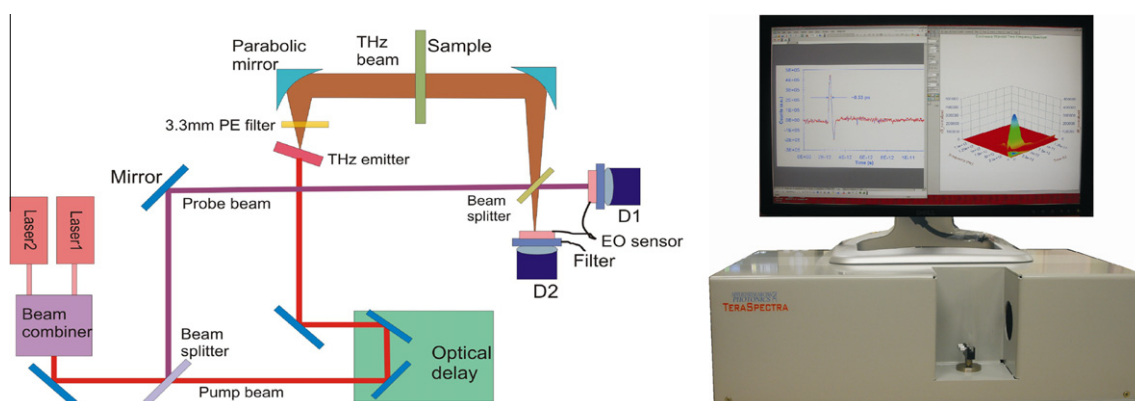


Fig. 1. Left: Layout of time-domain measurement system. Here two lasers are used with wavelengths 1064 nm and 945 nm, respectively. Consequently, a broadband terahertz spectrum is produced. Right: ARP's TeraSpectra© implementing the layout.

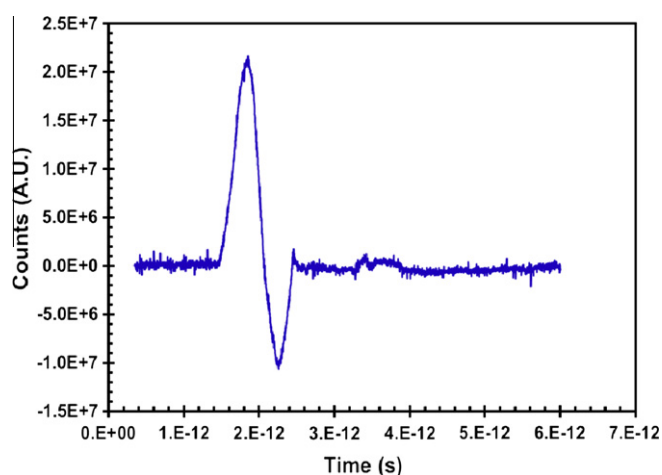


Fig. 2. Representative time-domain temporal signal (aka THz Pulse or interferogram) of the empty spectrometer.

within the first few terahertz, while over a longer range almost all molecules will exhibit discernible behavior. DFG enjoys particular advantage in this regard. Because the resulting terahertz frequencies are given by the difference of the pump laser frequencies, $\nu_{\text{THz}} \propto \nu_1 - \nu_2$, one can choose appropriate pump frequencies to obtain desired output terahertz bandwidth [3]. In practice, however, both ν_1 and ν_2 are not single frequency lasers because the diode lasers always have a bandwidth distribution around their main peaks; as such when a stationary beam of the generated terahertz is scanned by a moving beam derived from the same source, a wide frequency distribution will result. This is the so called electro-optic sampling or probing and the basis of the so called pump-probe method.

3. Terahertz time-domain spectrometer

Fig. 1 illustrates the functional diagram of a terahertz time-domain spectrometer¹ (THz-TDS) where the EO Dendrimer is used as the terahertz emitter as well as the sensor. Here we used two diode lasers at 1064 nm and 945 nm, respectively; consequently, a

¹ Although the term “spectrophotometer” has been used traditionally based on the fact that most spectrometer functions based on the measurements of light of one kind or another, however, the terahertz spectrometer deploys a different mode of measurements, viz., the time-domain measurements based on electro-optic sampling. Therefore, most authors in this area including this one choose to use the term “terahertz time-domain spectrometer (THz-TDS).”

range of ~ 35 THz is expected. A compact layout is achieved by arranging the components in the manner shown in Fig. 1. An interferogram is generated when the stationary beam is scanned by the terahertz beam whose intensity distribution is captured by the detector pair. Fig. 2 exhibits the time-domain pulse generated for the setup without any sample in. A self-calibrating algorithm is implemented such that the effect of atmospheric moisture is minimized. That is, a sample must be placed in the spectrometer for it to be measured; otherwise, the spectrometer will reproduce the same (empty) characteristic spectrum. A slightly different algorithm called Fourier transform of unevenly sampled data (aka Lomb periodogram, Fig. 3) [4] was deployed to analyze the experimental data. This algorithm essentially defines a transform by suppressing spectral leakage. Fig. 3 exhibits that the spectrum spans up to ~ 35 THz. In practical measurements this “empty” spectrum will serve as the background but when the sample is placed on a substrate (e.g., glass slide), then the blank substrate spectrum will serve as the background.

4. Principle of THz spectroscopy

When THz radiation interacts with molecules, it may stimulate many resonances such as molecular vibrations, phonons and/or other resonances in the system (in general molecular “events”), resulting in the THz photons being affected by characteristic amounts determined by a specific interaction or event (see Fig. 4). The change in energy and/or frequency yields information about the molecular nature of the interaction. Infrared and Raman spectroscopy yields similar information but not capable of detecting many resonant states as can be detected with THz because terahertz photons are sensitive to the vibrational states of the entire molecule as opposed to just a bond or charge state. Molecular simulation, especially molecular dynamics, reveals that there are numerous vibrational and conformational states possible when a molecule is not at its lowest energy state. As most material remains at its lowest energy state under normal and steady state conditions, THz perturbation will stimulate possible available states in the low frequency regions.

In common techniques such as Raman or IR spectroscopy, typically, a sample is illuminated with a laser beam. Light from the illuminated spot is collected with a lens and passed through a monochromator. Wavelengths close to the laser line, due to elastic Rayleigh scattering, are filtered out while the rest of the collected light is dispersed onto a detector. Modern Raman instruments employs notch or edge filters for laser rejection and spectrographs (either axial transmissive (AT), Czerny–Turner (CT) monochromator) or FT (Fourier transform spectroscopy based), and CCD

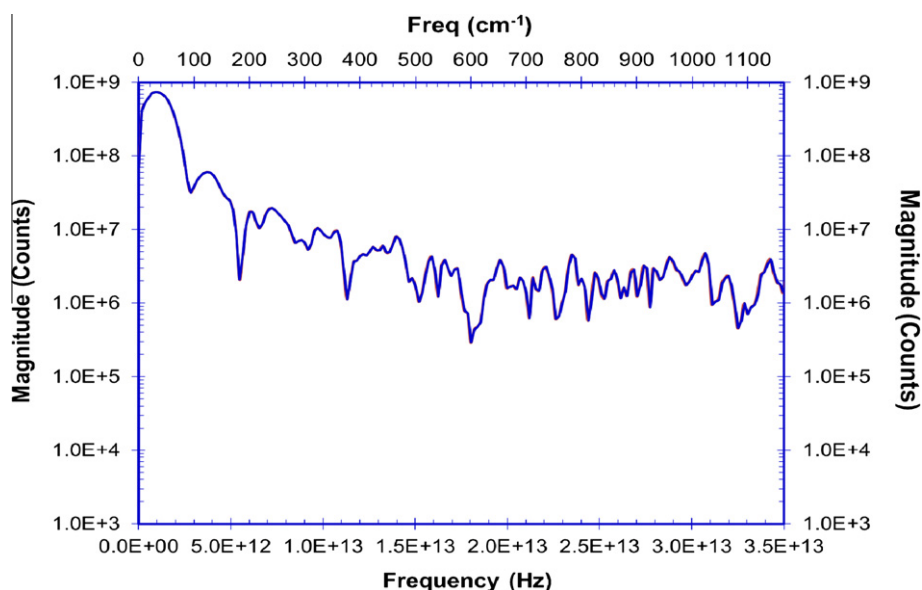


Fig. 3. Fourier transform terahertz magnitude spectrum of the THz pulse of Fig. 2.

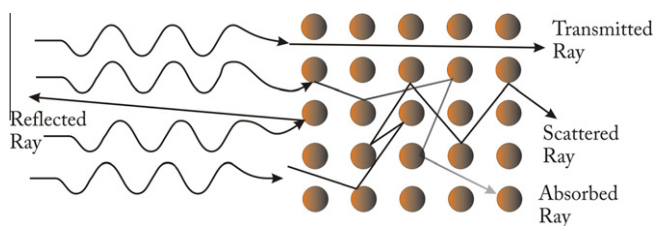


Fig. 4. Interaction of photons with matter; four different situations may occur: reflection, transmission, absorption and scattering. Both transmitted and reflected beam will include the scattered beam.

detectors. Spontaneous Raman scattering is typically very weak, as a result the main difficulty of Raman spectroscopy is in resolving the weak inelastically scattered light from the intense Rayleigh scattered laser light. This is a fundamental limitation of this method and though Raman gives more information than (e.g., IR spectroscopy), it is still not sensitive enough for resolving many states, especially the closely spaced ones.

Fig. 4 illustrates the interactions of radiation with a material system. The balls represent the lattice which may be composed of atoms for crystalline materials or of molecules (macromolecules) for amorphous and organic materials. Considering the lattice is composed of macromolecules, the material is most likely to be an amorphous matrix. In this case the transmitted beam will be composed mainly of the photons that have been involved in interaction with the matrix but not absorbed. Therefore, the transmitted beam will carry information about the material; and equivalently the reflected beam will also carry information about the nature of the matrix. Quantitative prediction of such information is obviously materials specific and best determined by experimental measurements.

5. Application examples

In this section, a few applications of the terahertz time-domain spectroscopy is described. In particular, molecular systems having very small differences have been considered where existing methods such as IR or Raman is not sensitive enough to discern closely lying events. Since terahertz interaction with molecules is sensitive to the vibrational modes of the entire macromolecule, THz-TDS has

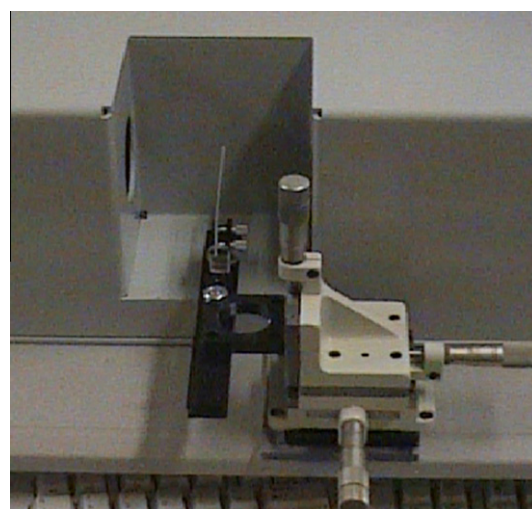


Fig. 5. Sample (spot) on a glass slide mounted on a XYZ-stage placed in the beam path.

been successfully applied to characterize a number of molecular phenomena such as non-ionic detergents in biopharmaceuticals [5], transdermal drug delivery [6], single nucleotide polymorphism [7], DNA hybridization [8], and molecular chirality [9].

5.1. Detection of explosives

Because of its specific molecular selectivity, THz-TDS is promising for explosive detection. Many packaging or concealment materials are semi-transparent at THz frequencies. Because terahertz can penetrate into many non-metallic containers, it is capable of identifying the explosive molecules within such containers. Because trace amount of explosives are present on these containers, a high sensitivity spectrometer may also be able to pick up explosive's signature from external traces as well. So, the problem is to understand how the THz interaction within target materials (by, for example, granular morphology) or near the target (by packaging) affects observation of characteristic resonances or vibrational

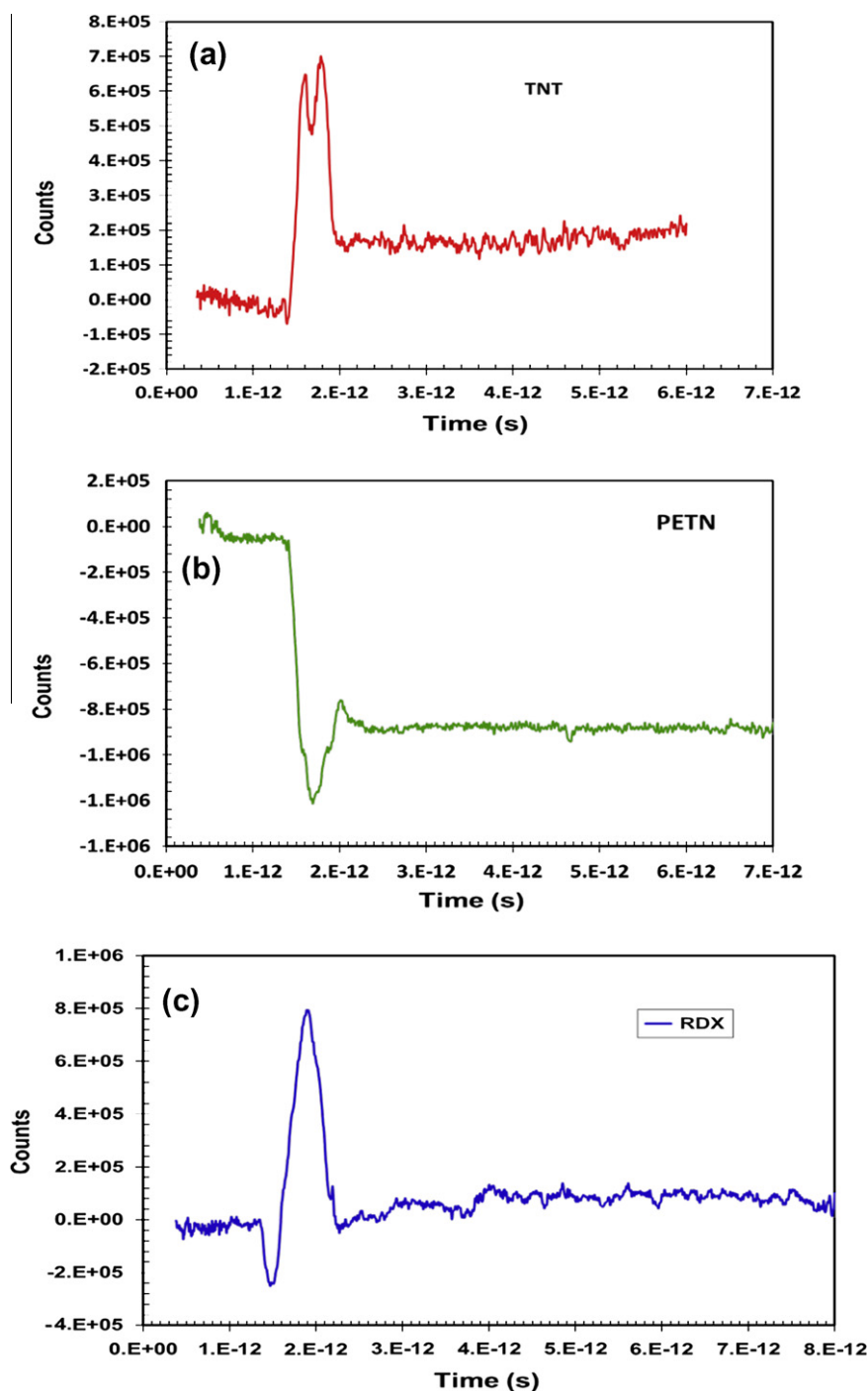


Fig. 6. Temporal signal (interferogram) of known explosive samples on glass slide (see insert): (a) TNT, (b) PETN, and (c) RDX.

states. Recently there has been a renewed interest to apply terahertz technology for explosives detection from various sources [10–14]. However, so far these attempts enjoy only partial success. A main reason is that the current art of terahertz spectroscopy allows collecting spectra over a narrow terahertz window, only up to $\sim 3\text{--}6\text{ THz}$. Within this range many compounds may look similar; this window is not enough to distinguish different explosives and their matrices uniquely. Wider terahertz bandwidth is expected to identify significant features in the acquired spectra specific to each molecule. Recently, we have demonstrated that molecular specimen having granular morphology with both smooth and rough surfaces can be uniquely characterized by the wide band terahertz spectroscopy [15].

A key factor in remote identification of the explosives is the molecular signature recognition of the compounds from trace amount of residues. To achieve higher success rate and low false alarm, the technology must have very high sensitivity to pick up the right signature from very small amount of material remaining in the residue. Such sensitivity may be exploited to incorporate selectivity of the explosive materials by generating their terahertz spectral signature and building a library. Such libraries will also be useful for other initiatives. Another task will involve the design and implementation of an appropriate protocol for testing and deploying the devices in the field. Since terahertz is capable of penetrating through fog and sand storm, this technology is an ideal candidate for remote identification of explosives.

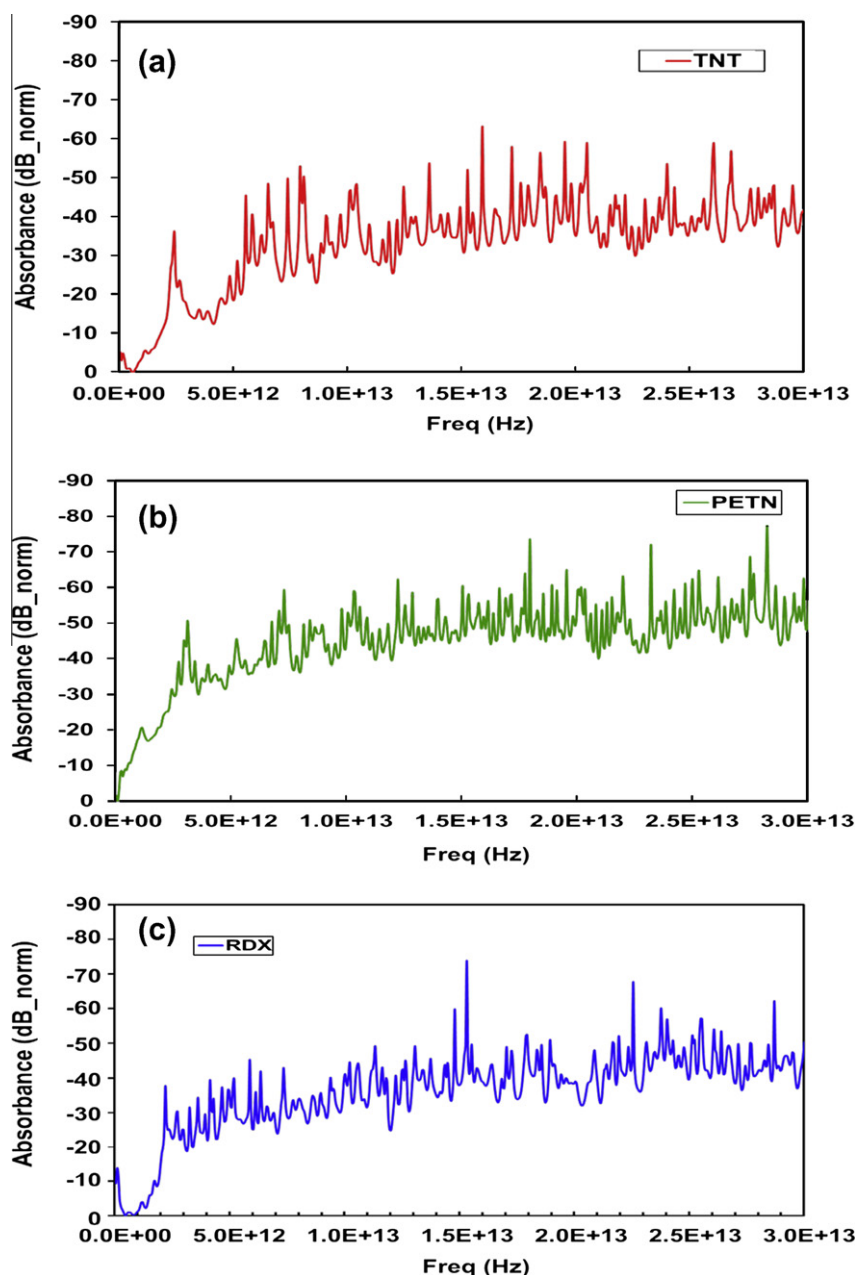


Fig. 7. Fourier transform absorbance spectrum of the three explosives over 0.1 THz to 30 THz: (a) TNT, (b) PETN, and (c) RDX.

Another key factor for extended range (distance) detection is the intensity of terahertz radiation. While EO Dendrimer technology allows generation of milliwatts of terahertz power [3], this may be further enhanced via a chip based terahertz generator. This involves fabrication of terahertz chip from EO Dendrimer that will enable terahertz amplification via waveguide technology [16].

5.1.1. Explosives' spectra

The most commonly used military explosives are pentaerythritol tetranitrate (PETN), a nitrate ester; cyclotrimethylenetrinitramine (RDX), a nitramine; and 2,4,6-trinitrotoluene (TNT), a nitroarene. The first two are white powders, but they may be plasticized. TNT can range in appearance from a creamy white powder to a yellow solid. Because TNT is melt-castable, it is often the matrix for PETN or RDX, e.g., Pentalite or Comp. B, respectively.

TNT, PETN and RDX samples were prepared as 1 mg/mL solution in methanol [17]. An aliquot (30 μ L) of each solution was

dispensed on a glass slide that was dried at ~ 45 $^{\circ}$ C for 10 min. Each glass slide with resulting spot was mounted on a XYZ-stage and placed in the beam path of a terahertz spectrometer (TeraSpectra, Applied Research and Photonics) such that the beam is vertical to the sample plane (see Fig. 5). Respective temporal signals were acquired in transmission mode. Fig. 6 shows the temporal signals (interferogram) of all three samples. As can be seen, each temporal signal is distinctively different in both magnitude and shape, thus exhibiting unique features for identifying the compound. Fig. 7 shows the Fourier transform absorbance spectra of the respective samples over a range of ~ 0.1 THz to 30 THz. Here also the spectra exhibit distinct features for each compound. Within each spectrum there exist distinct absorption peaks that can be used to identify the species.

Since terahertz radiation is sensitive to the vibrational states of the entire molecule (as opposed to a bond vibration or its rotation), the absorbance peaks in Fig. 7 correspond to the vibrational states

of the molecules of the present investigation. However, further investigation is necessary to establish a relationship between the significant peaks with the molecular structure. Nevertheless, these initial spectra are promising and indicate the feasibility for further investigation within this region of rich spectral activities. For example, the observed trend in the RDX spectrum (Fig. 7c) matches with those reported within the first 4 THz (see Ref. [18], Fig. 5e).

5.2. Vibrational states of C_{60} and $H_2@C_{60}$

Here we describe another example of the vibrational states of two Fullerenes, C_{60} and $H_2@C_{60}$. The two molecules differ only by two Hydrogen atoms from one another and thus difficult to distinguish their spectral features using standard spectroscopy methods, e.g., see Ref. [19]. Here, THz-TDS was used to determine the features in the respective spectra of the above two Fullerenes.

Pure C_{60} and $H_2@C_{60}$ were received in powder form. The samples were kindly provided by Dr. Nicholas J. Turro's Lab from Columbia University, NY. Two solutions were prepared in 1, 2-dichlorobenzene (solvent) via gravimetric method. C_{60} : As received 5.09 mg C_{60} (solute) was added to 3.33863 g solvent, yielding a solution of 1.52458 mg/g. $H_2@C_{60}$: As received 2 mg $H_2@C_{60}$ (solute) was added to 1.30954 g solvent, producing a solution of 1.52725 mg/g. Thirty micro liter of each solution (equivalent to $\sim 45.818 \mu\text{g}$ solid) were dispensed on two glass slides and the resulting drops were allowed to dry on a hot plate at 45°C for ~ 15 min. The slides were mounted on a XYZ stage one at a time and their spectra were acquired.

5.2.1. Results and discussion

Fig. 8 shows the time domain temporal signal of both samples. The $H_2@C_{60}$ pulse shows lower peak height compared to pure C_{60} as well as different peak positions. Fig. 9 exhibits the Fourier transform absorbance spectra of the two Fullerenes obtained from their respective pulses that show distinctively different peaks compared to each other. Table 1 summarizes the absorbance peaks for both C_{60} and $H_2@C_{60}$ extracted from their spectra and also those obtained from IR spectra [19]. As seen from Table 1, there is a number of absorbance peaks present in the THz spectra that were not detected by the IR spectra. This is indicative of the sensitivity obtainable from THz interaction with the entire molecule. That is as the molecular conformation change as a function of time-dependent stimulus on the femto- to pico-second scale (or, equivalently, the frequency of the stimulus on the THz scale), the absorbance is directly probed and recorded by the detection system. While some far lying states (e.g., 4250 cm^{-1}) were observed by low-temperature (6 K) IR spectroscopy and explained by translational and rotational motions of H_2 inside C_{60} cage [20], those studies were also limited to the detection of only a few number of states. The observation of multiple states in the low frequency region indicates that the vibrational states of these molecules can be effectively probed by THz.

It has been predicted by Turro et al. [21] that an important ability of the Fullerene research is the possibility of controlling the spin selectivity of the catalyzed conversion of the para species, $pH_2@C_{60}$, into the ortho species, $oH_2@C_{60}$, so that a strong nuclear spin polarization may be produced. However, if spin selective $H_2@C_{60}$ is produced, it seems to be difficult for standard IR spectroscopy to easily distinguish and identify the ortho and para states. The presence of distinguishable absorbance peaks identified by THz spectra suggests that this tool has the required sensitivity for detecting the spin isomers of H_2 inside C_{60} . Additionally, the presence of additional peaks indicates that this method can detect modes not visible in standard IR, yielding unique insight into uncharacterized host/guest interactions.

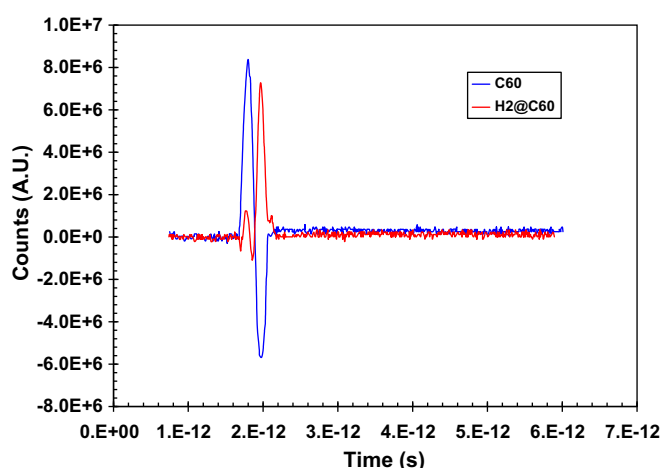


Fig. 8. Time domain signal of C_{60} and $H_2@C_{60}$ exhibit clear difference between C_{60} and $H_2@C_{60}$ pulses.

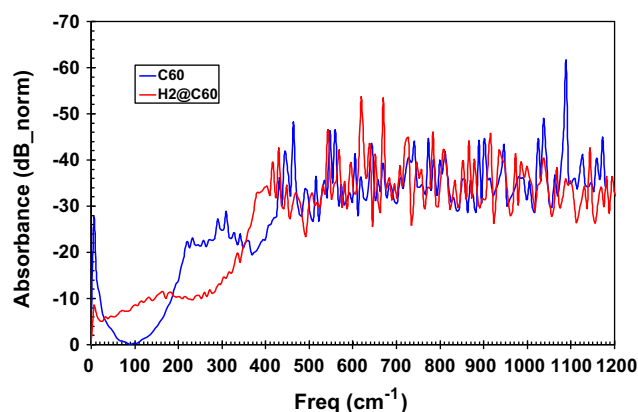


Fig. 9. Fourier transform frequency spectra of C_{60} and $H_2@C_{60}$ show distinct absorbance characteristics.

Table 1

Comparison of IR and THz spectra of C_{60} and $H_2@C_{60}$. All units are in cm^{-1} .

C_{60} : THz [present study]	C_{60} : Ref. [23]	$H_2@C_{60}$: THz [present study]	$H_2@C_{60}$: Ref. [23]
6.44, 219, 232, 258, 271, 290	–	6.32, 164, 183	–
309, 328, 341, 361, 393	–	335, 379	–
406, 432, 444, 464, 490	–	404, 417, 430, 449, 468	–
515, 535, 543, 560, 593	526.6, 576.7	506, 543, 569, 581, 594	526.5, 576.7
605, 618, 644, 670	–	619, 638, 651, 670	–
740, 772	–	708, 727, 746, 784	–
857, 889	–	809, 834, 853, 866, 878	–
902, 947, 992	–	916, 935, 973, 986, 998	–
1024, 1037, 1088	–	1036, 1068, 1087	–
1127, 1159, 1172	1182.3, 1429.2	1131, 1144, 1169, 1182, 1194	1182.3, 1429.2
Total: 38	4	41	4

So an obvious question is, “are the additional states observed in Fig. 9 (or, Table 1) real?” Menendez and Page [22] have illustrated various vibrational states assignment of C_{60} from theoretical calculations [23]. These authors have pointed out a number of vibrational states present in C_{60} along with their multiplicities. Table 2 summarizes the predicted states of C_{60} (col. 2) and also the measured states of the present study (col. 1). Modes whose frequencies

Table 2

Comparison of IR and THz spectra of C₆₀ and H₂@C₆₀. All units are in cm⁻¹.

C ₆₀ : THz [present study]	C ₆₀ : Ref. [23]
6.44, 219, 232, 258, 271, 290	272
309, 328, 341, 361, 393	343, 353
406, 432, 444, 464, 490	403, 433, 485, 496
515, 535, 543, 560, 593	526, 534, 553, 567, 568, 575
605, 618, 644, 670	668
740, 772	709, 736, 743, 753, 756, 764, 772, 776, 796
857, 889	831
902, 947, 992	961, 973, 984
1024, 1037, 1088	1079, 1099
1127, 1159, 1172	1182
Total: 38	30

appear in bold type (Table 2, col. 2) have been unequivocally identified from Raman, IR, neutron, or fluorescence experiments [23]. Moreover, each of the states enumerated in col. 2 have associated degeneracies; e.g., the state at 272 cm⁻¹ has a degeneracy of 5 and so on [23]. So some of the observed states of the present work potentially could be due to the degeneracy of the states as indicated by Ref. [23]. However, further investigation is necessary to establish whether or not the differences observed in the respective spectra of C₆₀ and H₂@C₆₀ are due to the presence of H₂ in C₆₀ or if some other artifact is responsible. For example, the addition of the spectrum of D₂@C₆₀ would be important to see if there is a rational pattern. This is being planned for a continuation of the present work. Therefore, the main inference of the present investigation is that the terahertz spectra clearly shows difference between C₆₀ and H₂@C₆₀, however, further work is necessary to establish clear evidence that H₂ is actually causing the spectral differences or not. Other possibilities such as whether different morphologies of the two samples or different levels or types of impurities are responsible for the observed differences should be considered in future work.

In the same way, the three explosives present very different terahertz signals, but a question arises that if other non-hazardous materials would generate similar or identical spectra, thus rendering the detection ambiguous. To this end our observation is that, of all the molecular systems we have studied so far [5–9], no two molecule exhibit exactly identical absorbance peaks. This is primarily due to two prominent features of the underlying technology: (1) very high sensitivity available from the TeraSpectra down to parts per trillion [8] and (2) a wide window over 0.1 THz to ~35 THz. There are many molecular systems, especially the ones having close molecular weight, may exhibit similar trend within a narrower window of observation (e.g., 0–3 THz), but over a broadband window of observation of the present study, the difference between two molecules even of very close molecular weight become clear.

Thus, it is evident that the THz-TDS of the present work provides a suitable tool to continue both theoretical and experimental work with the vibrational states of the Fullerenes and other molecular system.

6. Summary

Broadband terahertz radiation has been generated from electro-optic Dendrimer via difference frequency method (two-photon excitation). A sharp time-domain pulse is obtained by standard electro-optic sampling method of detection. The Fourier spectrum obtained from the time-domain pulse spans up to ~35 THz. This

wider THz range is expected to aid in high sensitivity characterization of molecular interactions because terahertz radiation is sensitive to the vibrational states of an entire molecule (as opposed to bond or torsional vibrations, or charge state that are usually probed by other methods). Two examples of application have been outlined. It has been shown that common explosive traces can be uniquely identified by their THz spectra. It is also shown that the vibrational states of two Fullerenes having very close molecular weights can also be uniquely probed for their vibrational states.

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