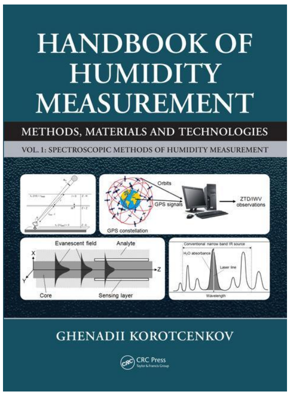

Handbook of Humidity Measurement Methods, Materials and Technologies: Spectroscopic Methods of Humidity Measurement

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Atmosphere Monitoring Using Methods of Absorption of Electromagnetic Radiation—Terahertz Absorption

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7 Atmosphere Monitoring Using Methods of Absorption of Electromagnetic Radiation—Terahertz Absorption

7.1 TERAHERTZ RADIATION

Terahertz (THz) radiation is the electromagnetic spectrum with the frequency defined from 0.1 to 10 THz (1 THz = 10^{12} Hz). This spectral range corresponds to a wavelength between 30 μm and 3 mm. THz frequencies lay between the operation ranges of classical microwave and infrared spectroscopy (Figure 7.1) and thus cannot be effectively covered by any of these techniques. However, THz region plays an important role for the Earth's radiation budget; for example, (1) up to 50% outgoing long-wave radiation (OLR) is below 650 cm^{-1} and (2) up to 50% of basic greenhouse effect is in THz/far-IR range. But, until recently, because of the difficulty of generating and detecting techniques in this region, THz frequency band remains unexplored compared to other range and tremendous effort has been made to fill in *THz gap* (Zhang and Xu 2009). Significant progress in the development of THz region has been achieved only in the last 30 years, when suitable for use sources and detectors of THz radiation have been developed (Gallerano and Biedron 2004; Krishna et al. 2012; Yin et al. 2012; Lewis 2014).

7.2 ABSORPTION OF THz RADIATION

Currently there are large amounts of research related to the study of the propagation of THz radiation in the atmosphere. But the main efforts are focused on the study of the atmospheric transparency in the THz range and searching the air transmission windows for communicating and sensing system (Yuan et al. 2003; Foltynowicz et al. 2005; Yang et al. 2011, 2012; Yao et al. 2012). As is known, THz communication will benefit from the high-bit-rate wireless technology which takes advantage of higher frequency and broader information bandwidth allowed in this range than microwave. It is possible for such a system to achieve data rate in tens of gigabits per second (Lee 2008). However, the atmospheric opacity severely limits the communication applications at this range (Siegel 2002). Water vapor in the air has a high absorption coefficient in the THz frequency range. The humidity affects the shape and amplitude of pulsed T-rays, especially in long distance propagation. Water vapor absorption lines are extremely ubiquitous and result in relatively short atmospheric propagation paths through most of the THz range. In addition, as the relative humidity increased, the *windows*, which present on the THz range, got narrower and shallower. Therefore, the definition of conditions for THz radiation propagation over long distances will undoubtedly determine whether THz communication will be carried out into practical application. That is why the characteristics of THz atmospheric propagation now rank among the most critical issues in the principal application of space communication and atmospheric remote sensing (Tonouchi 2007).

7.3 ATMOSPHERE MONITORING USING THz SPECTROSCOPY

Research has shown that the absorption in THz region is due mainly to intense rotational transitions of the water molecules (Hall and Dowling 1967; Burch 1968; van Exter et al. 1989). In gases like water vapor, the relative isolation of the molecules leads to sharp resonant peaks of absorption centered at specific frequencies (Figure 7.2). The intensity of these peaks is strongly dependent on the concentration of water vapor in the atmosphere. Furthermore, the absorption strength of water vapor lines is proportional to the concentration of water molecules, and therefore to the relative humidity. This means that THz spectroscopy can be applied for measurement of atmospheric humidity (Xin et al. 2006). However, despite the encouraging prospects, it should be recognized that there is no significant progress in the use of THz radiation for monitoring the atmosphere.

Unfortunately, despite the significant progress in theoretical predictions of atmospheric absorption

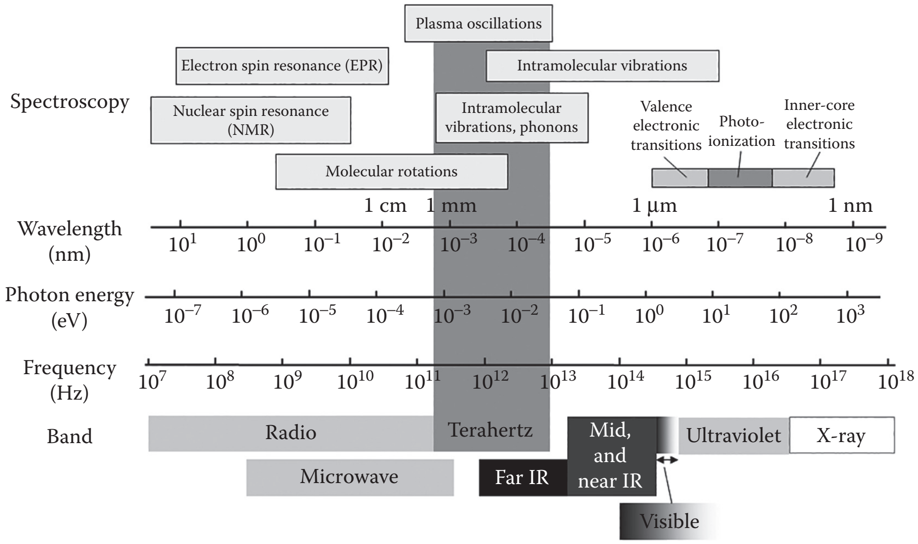

FIGURE 7.1 Electromagnetic spectrum from radio to X-ray wavelengths, including spectral ranges for probing different excitations in materials.

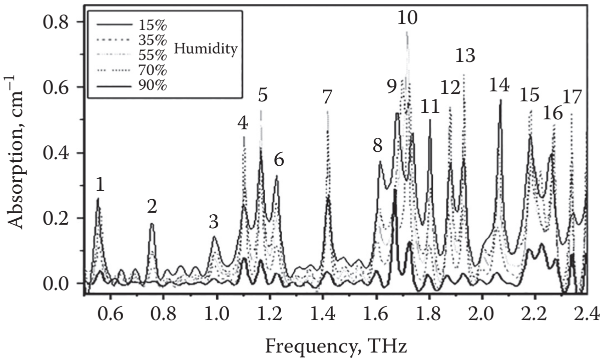

FIGURE 7.2 Water vapor absorption profiles at different humidities at room temperature. Air with a relative humidity of 10%, 30%, 50%, and 90% used in these experiments. (Reprinted with permission from Xin, X. et al., *J. Appl. Phys.*, 100, 094905, 2006. Copyright 2006 American Institute of Physics.)

(Liebe 1989; Rosenkranz 1998; Pardo et al. 2001b), we did not yet reach a good understanding of the behavior of THz-wave in the atmosphere, and the knowledge and technology for the atmospheric remote sensing are still poor. There still exist large difficulties in developing appropriate observation technology for THz-wave that lies in the transition region between optical and microwave regions.

As a result, now we do not have yet an adequate model that could describe the atmospheric propagation THz radiation in different kinds of climatic conditions. Therefore, in order to develop hygrometers, working in THz region, and on the basis of their readings to make predictions on the climate change, we need a detailed studies aimed at (1) the construction of radiative transfer algorithm; (2) the collection of accurate spectral parameters, such as linear and continuum absorption and complex refractive index in THz region, that is, to collect the spectroscopic fingerprinting of atmospheric molecules for THz atmospheric monitoring; and (3) the standardization of measurement procedures with purpose to improve both the signal-to-noise ratio (SNR), and the restoration of the original signal from the observed signal by the process of deconvolution (Foltynowicz et al. 2005; Ryu and Kong 2010; Zhan et al. 2015). It is essential to understand the actual effects on the amplitude and phase of THz radiation propagating through the atmosphere, which depends on the frequency of incident wave, gas components, and ambient temperature or barometric pressure in different atmospheric conditions (Yao et al. 2012).

Xin et al. (2006) have found that the line amplitude variations of three orders of magnitude can be accurately measured with THz spectroscopy, providing a large dynamic range for humidity measurements. However, the lines used for humidity measurement must be carefully selected and the variation of their amplitude with humidity must be calibrated. As it is seen in Figure 7.3, the influence of humidity strongly depends on the spectral range used for measurements.

It should also be borne in mind that to build the correct model the propagation of THz radiation in the atmosphere, more completed information is needed on the absorption spectra of water vapor. Three spectroscopic parameters: (1) center of the line, (2) oscillator intensity,

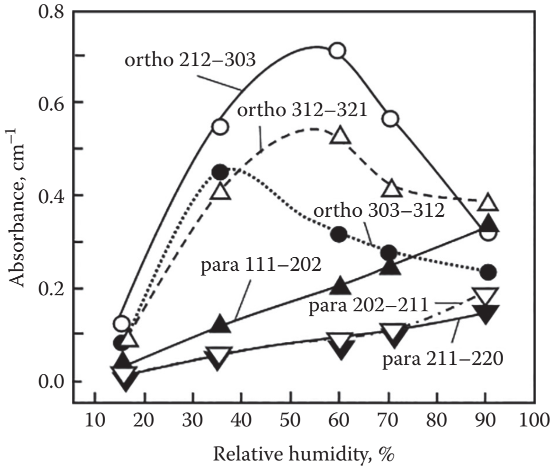

FIGURE 7.3 Color online the peak intensity of three para triangles and three ortho squares rotational transitions for different humidities. While para transitions increase, ortho transitions increase and then decrease with increasing humidity. (Reprinted with permission from Xin, X. et al., *J. Appl. Phys.*, 100, 094905, 2006. Copyright 2006, American Institute of Physics.)

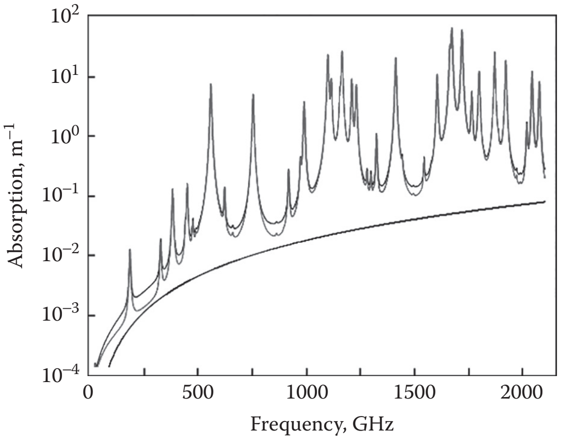

FIGURE 7.4 The calculated air-broadened spectrum (light gray), continuum absorption (black), and air-broadened spectrum with continuum absorption (gray). All plots are 15.23 Torr of water vapor and 746.77 Torr of air. (Reprinted from *J. Quant. Spectrosc. Rad. Transfer*, 127, Slocum, D.M. et al. (2013) Atmospheric absorption of terahertz radiation and water vapor continuum effects, 49–63, Copyright 2013, With permission from Elsevier.)

and (3) pressure broadening coefficient fully describe the absorption lines (Yasuko and Takamasa 2008; Rothman et al. 2009). While the first parameter is well determined for all of the lines because it depends only on the molecule inner structure, the other two are much more difficult to predict because they depend on the molecule interactions. It has been known for a long time that the line contribution is not enough to reproduce the experimentally measured atmospheric absorption, with calculated absorption being as much as an order of magnitude too small. In order to explain the discrepancy, the so-called continuum absorption was introduced (Rice and Ade 1979; Ma and Tipping 1990; Pardo et al. 2001a). The atmospheric absorption spectrum doesn't correspond to the accumulation of water vapor absorption lines. The continuum absorption is what remains after subtraction of linear contributions from the total absorption that can be measured directly (Rosenkranz, 1998) (Figure 7.4). It may be observed in wide electromagnetic spectrum (from microwave to infrared) and cannot be described by water vapor absorption lines. The physical mechanisms of the continuum absorption are still not well explained while several theories have been proposed (Burch and Gryvnak 1979; Clough et al. 1989; Liebe 1989; Ma and Tipping 1992; Yasuko and Takamasa 2008; Yang et al. 2014).

Certain difficulties connected with the interpretation of the results are also related to the fact that the absorption cross section for H_2O vapor depends nonlinearly on the pressure and temperature leading to an absorption profile that can skew from a Beer–Lambert–Bouguer model with respect to environmental conditions (Xin et al. 2006). It was also established that the absorption spectra for water vapor are a mixture of the contribution of two components, *para*-H_2O and *ortho*-H_2O with an *ortho* to *para* ratio (OPR) of 3:1, which even more complicates their structure. *Para*-H_2O and *ortho*-H_2O appear when considering the nuclear spin effect of hydrogen atoms in the water molecule (Townes and Schawlow 1975). At that Xin et al. (2006) have shown (Figure 7.3) that water molecules that undergo *ortho*-level rotational transitions increase, then decrease in absorption strength with increasing humidity, whereas most *para* transitions simply increase as expected for higher concentrations in the THz beam path. In terms of nuclear spin statistics, the *ortho* levels possess populations three times larger than the *para* levels at room temperature due to the presence of two symmetric hydrogen nuclei.

It is not clear yet how can we separate desired signals from the thermal blackbody-background created by any warm object present in the THz (Foltynowicz et al. 2005). Research has shown that background is a severe issue in this frequency range, much more so than for other frequency ranges due to blackbody radiation. The spectral density of background radiation from any room temperature object increases at the long wavelengths by about four orders of magnitude per decade. Since this is so prevalent, if we wish to get clean signals, one has to think about coherent detection, very narrow line-width sources, and very narrow line-width detectors, to minimize saturation of the detector.

In addition, one should take into account the technical immaturity of the technology in this frequency range, including the lack of compact, tunable sources, detectors, and other standard optical components (Foltynowicz et al. 2005), which restrains the development of devices suitable for wider application.

Furthermore, it should be noted that the measurements themselves in the field of THz are not trivial. For example, THz spectroscopy operates with pulses of electromagnetic radiation which are too short to be resolved by conventional electronic display instruments such as oscilloscopes. Usually, for research in this area it is used so-called the transmission time-domain THz spectroscopy (TTDTS). The basic idea of the TTDTS can be described in the following way: a sub-picosecond pulse of electromagnetic radiation passes through a sample and gets its time profile changed compared to the one of the reference pulse. The last can be either a freely propagating pulse or a pulse transmitted through a medium with known properties. Through an analysis of changes in the *complex* Fourier spectrum, which are introduced by the sample, the spectrum of the refractive index of the sample's material is obtained.

TTDTS is being realized by so-called gated-detection technique (Khazan 2002). Typical gated-detection scheme usually used for studies is shown in Figure 7.5. The THz spectrometer is powered by a laser which emits a train of pulses each of several tens of femtoseconds (1 fs = 10^{-15} s) in duration. The initial laser beam is split by a beam splitter in two parts called pump and probe beams. A pump pulse hits an emitter which in response releases a short (few picoseconds) pulse of electromagnetic radiation. The spectrum of the radiation is centered at several hundreds of GHz (1 GHz = 10^9 Hz) so that one- or even half-cycle pulses are released. This THz radiation then comes to a detector which is gated by a probe pulse. The output signal of the detector is proportional to the magnitude and the sign of the field of the THz pulse in every certain moment of time. Thus, by the variation of the delay between pump and probe optical pulses one can trace the whole time profile of the THz pulse.

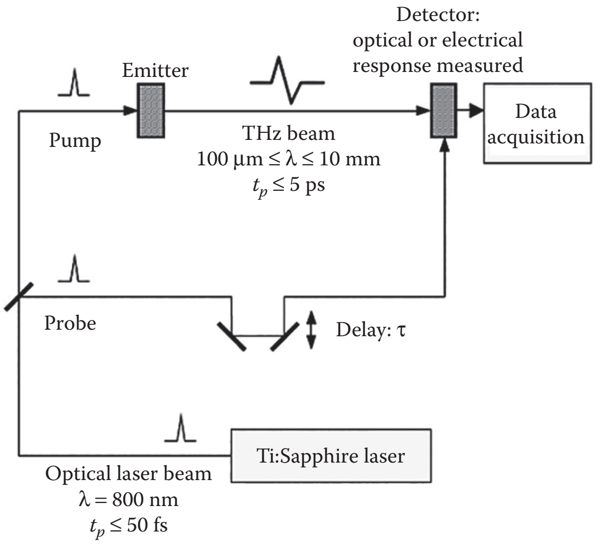

FIGURE 7.5 Typical gated-detection scheme: λ is wavelength and t_p is pulse duration (of either laser or THz pulse). (Idea from Khazan, M.A., Time-domain Terahertz spectroscopy and its application to the study of high-T$_c$ superconductive this films, PhD Thesis, Universität Hamburg, Germany, 2002.)

Main sources and receivers of THz radiation, which can be used in the development of devices suitable for monitoring the atmosphere, are listed in Tables 7.1 and 7.2. Their advantages, shortcomings, and opportunities for the applications are considered in detail in Gallerano and Biedron (2004), Krishna et al. (2012), Yin et al. (2012), and Lewis (2014). For example, large scale free electron lasers (FEL) facilities are capable of providing a high power *continuous-wave* (CW) THz radiation while low-cost solid state sources are expected to drive the realization of more portable THz systems for a variety of applications (Gallerano and Biedron 2004). According to Yin et al. (2012) most advanced solid state T-ray sources which are primarily used for current popular THz research include (1) pulsed T-rays based on ultrafast laser sources; (2) high-frequency electronic sources, for their integration into existing electronic technology, to achieve low-power *continuous-wave* (CW) operation; and (3) *Quantum Cascade Laser*s (QCLs), for their small size and tunability, to realize CW operation. The pulsed nature of ultrafast T-ray systems provides high SNRs, broad bandwidth, and low average power, making them ideal tools to study biological and medical materials. However, QCLs as THz narrowband laser sources show deeper penetration, due to higher average power, which complements Tuned Port Injection (TPI) systems.

As for the receivers of THz radiation, there is also no definite opinion about what receiver is preferred for use. Everything is determined by the radiation power and frequency range. So, traditionally used in the microwave and IR radiation detectors that operate at temperatures close to room temperature (piezoelectric detectors, bolometers, thermocouples, etc.), have good sensitivity in the THz range, but their use in the THz systems is limited by low operation speed which at best is tens of milliseconds.

Detectors based on the Schottky barrier diodes perform significantly better frequency properties. For example, Microtech Inc. (USA) manufactures detectors based on GaAs, which can operate in the range of 0.2–1.0 THz. The

TABLE 7.1
Some Sources of Terahertz Radiation

Source Type	Example
Thermal	Cosmic background radiation
	Globar
	Mercury lamp
Vacuum electronic	Backward-wave oscillator
	Extended-interaction klystron
	Traveling-wave tube
	Gyrotrons
	Free electron lasers (FELs)
	Synchrotrons
Solid-state electronic	Gunn diodes
	Transistors
	Frequency multiplication
	Superconductor
	Resonant tunnel diodes
Lasers	Molecular gas lasers
	Ti:Sapphire-based Lasers
	Semiconductor lasers (*p*-type Ge laser)
	Quantum cascade lasers
Sources pumped by lasers (continuous)	Photomixer
	Mechanical resonance
Sources pumped by lasers (pulsed)	Photoconductive switches
	Air
	Magnetic dipoles
	Terahertz parametric oscillator
Optical rectification	Bulk electro-optic rectification
	Nonresonant optical rectification
Transient currents	Diffusion
	Drift
Mechanical excitation	Peeling tape
	Surface formation

Source: Data extracted from Lewis, R.A., *J. Phys. D: Appl. Phys.*, 47, 374001, 2014.

TABLE 7.2
Detection of Terahertz Radiation

Detector Type	Example
Electronic	• Schottky diode • Backward diode • Rectifying transistor (Tera-FET) • Superconductive mixers
Thermal	• Bolometer • Golay cell • Pyroelectric • Thermopile
Optical	• Photoconductive switches or antenna/photomixers • Electro-optic sampling • Biased-air coherent detection • Synchronous and asynchronous optical sampling

Source: Data extracted from Yin, X. et al., *Terahertz Imaging for Biomedical Applications: Pattern Recognition and Tomographic Reconstruction*, Springer Science+Business Media, New York, pp. 9–26, 2012, and from Zouaghi, W. et al., *Eur. J. Phys.* 34, S179–S199, 2013.

main advantages of these detectors are speed of response (up to 20 GHz), and operation at room temperature.

However, their sensitivity (10^{-9}–10^{-10}) is an order of magnitude worse than the sensitivity of superconducting receivers such as hot electron bolometer (HEB). Their work is based on the effect of heating of electrons in thin films of superconductors, when they absorb electromagnetic radiation. This means that the superconducting receivers are preferable when detecting very weak signals.

At the same time, experience shows that when working with a pulse radiation, it is better to use detectors based on electro-optic sampling and photoconductivity switch antenna/photomixer. There are reports that as the receivers, a quantum dots-based single electron transistors can also be used. However, their use is limited because the operating temperature is close to zero, 0.3–1.5 K (Lewis 2014).

One should realize that optical components such as mirrors, lenses, and polarizers are also components of THz spectrometers (Lewis 2014). In contrast to visible optical systems, where lenses and similar transmitting elements predominate, THz systems tend to employ reflecting elements, which have minimal loss and no dispersion. THz mirrors have conventionally been made of metal. Other materials have been recently trialed, for example, doped and undoped GaAs and a hybrid of polypropylene and high-resistivity silicon. Tunable mirrors, based on one-dimensional photonic crystals, have also been developed. Lenses are typically made of plastics. It is advantageous if the lens transmits visible radiation, as this facilitates optical alignment. Traditionally, plastic THz lenses were made by machining on a lathe. Recently, lenses have been manufactured by compressing various micropowders in metal moulds using a tabletop hydraulic press. Diffractive elements, cavities, and waveguides for THz range have also been constructed using micromachining technology. On the other hand, temporary, or reconfigurable, components may be formed by optical modulation, using visible light projected onto a silicon chip; aperture arrays and polarizers have been made in this manner.

As the TDTS is a new measurement technique, today's market of scientific instruments cannot offer a reasonably priced time-domain THz spectrometer. As a rule, such systems throughout the world are normally hand-made and are a subject of constant development and improvement. Therefore, modern plants designed to operate in THz region are quite expensive and their operation requires highly skilled professionals.

Figure 7.6 shows a diagram of the ZnTe crystal-based THz–TDS spectrometer used in Sandia Lab for investigations in THz range. This is photomixer-based spectrometer. The maintaining the stability of the entire system, including the ultrafast laser pulse shape, repetition rate, and power in the THz pumping and THz sampling beams, the generated THz pulse shapes, bandwidth and power, coupling into and out of the sample tube, and the temperature of sample is also important problem during experimental studies (Yang et al. 2011).

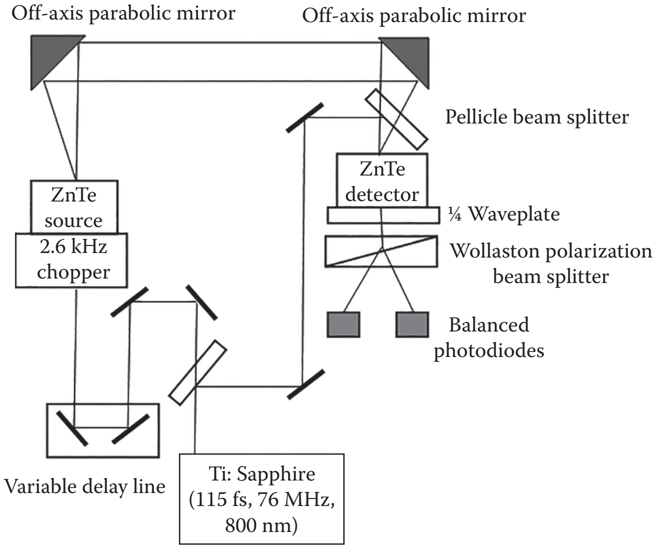

FIGURE 7.6 Terahertz time-domain spectroscopy overview. A mode-locked, Ti:Sapphire oscillator (Coherent Mira 900) with a 115 fs pulsewidth, 76 MHz repetition rate and centered at 800 nm is split into a high energy pump and low energy probe beam. The THz beam that is generated on the output face of the ZnTe crystal is collected and collimated by a 10 cm focal length, off-axis parabolic mirror with a 90° reflection angle. The THz beam traverses a path length of 94 cm and is focused onto a 10 × 10 × 0.9 mm thick <110> ZnTe crystal sensor. The polarization change is analyzed by allowing the probe beam to traverse through a ¼ wave-plate oriented at 45°. The polarization components are split by a Wollaston prism with a 10° divergence angle and the resulting split beam is weakly focused onto a large area balanced photoreceiver (New Focus Model 2307) with a gain setting of 10^5 V/A. The average probe optical power of each polarization component present at the balanced photodiode is 3.8 mW. The THz generation is modulated at 2.6 KHz and the differential signal is measured by a SRS Model 830 DSP lock in amplifier with an integration time of 300 ms and a low pass filter roll off of 12 dB/Oct. The data acquisition is controlled by LabVIEW 6.0 on a desktop PC. (From Foltynowicz, R.J. et al., Atmospheric propagation of THz radiation, Sandia National Laboratories, Report SAND2005-6389, New Mexico, 2005.)

The effect of atmospheric turbulence also needs to be taken into account. In parallel, scattering effect also results in the energy attenuation along the optical path. It comprises the molecular Rayleigh scattering and the Mie scattering by aerosols and the water vapor coagulum. As the wavelength of THz radiation lies in the order of aerosols, only Mie scattering should be taken into consideration. Aerosol particles mainly refer to the solid and liquid particles suspending in the atmosphere, for example, dusts, salts, ice particles and water droplets, and the Mie scattering effect mainly depends on their size-distribution, complex refractive index, and the wavelength of incident radiation. It is difficult to simulate the scattering by aerosols due to their large scale change in time and space domain.

This means that to build a theoretical model able to give information about the state of the atmosphere, basing on data of THz spectroscopy, an additional study of THz atmospheric transmittance as a function of water vapor content is required. However, data of these types are very limited in the open literature. Only a few broadband studies have been performed in the THz region, most of which were performed in a laboratory environment using either pure nitrogen or oxygen gas as the foreign broadening gas (Podobedov et al. 2005, 2008; Slocum et al. 2015). This method allows for control over the experimental conditions at the expense of using a foreign gas that is not of the same composition as the Earth's atmosphere. Other studies have been performed using atmospheric air (Pardo et al. 2001a, b), however these studies lack of control of the experimental parameters and are often performed at remote high altitude locations. No doubts that new technologies and approaches that will develop instruments suitable for wide application, are required (Krishna et al. 2012).

After solving all problems, the THz remote sensing technology can certainly be a powerful tool for the accurate prediction of the natural disaster, such as localized torrential rain and extraordinarily heavy snowfall, due to the climate change, because THz-radiation is suitable to observe humidity, ice cloud, and the temperature simultaneously with high spatial resolution (Liebe et al. 1993; Yasuko 2008). On account of the fact that water is highly absorptive in the THz range, while most materials which absorb moisture are either very transparent (e.g., paper, plastics) or reasonably transparent to THz, the THz spectroscopy can also become very prospective method for

THz imaging: a high contrast in the image between *moist* and *dry* regions could be a good tool for detecting materials with different properties (Federici 2012). Experimental studies confirmed this statement. THz imaging is shown to be a viable nondestructive evaluation tool not only for point measurements of water content, but also as a tool to visualize the spatial dependence of moisture.

Bidgoli et al. (2014) have shown that THz spectroscopy also can be successfully used for technological process control. For water vapor monitoring they used frequency scan in the range of 300–500 GHz (0.3–0.5 THz). The choice of this range ($f < 1$ THz) Bidgoli et al. (2014) explained by the fact that in this spectral range an absorption and scattering by the dust and tar components in the industrial gases are much less as compared to the THz waves at above 1 THz. In addition, within this spectral range, two strong rotational lines, at 448 and 383 GHz, have the potential to determine the concentration of water in gaseous mixtures. Other stronger lines (i.e., at 556 GHz) have a tendency to saturate at the high water vapor concentrations. The CO lines at 346 and 461 GHz can be used to quantify the concentration of CO in a complex gas mixture. A compact THz transmitter with an output power of 50–100 μW launched the EM waves into the gas-cell. Bidgoli et al. (2014) used a frequency multiplier-based Tx unit (Virginia Diodes, Inc., USA), which up-converted the microwave signal (8–14 GHz) to the desired range for this application (300–500 GHz). The frequency of the microwave source was controlled by a DC source that was integrated into a lock-in amplifier (Stanford Research SR830). The THz wave was amplitude-modulated (AM) at 18 Hz. To minimize the effects of instabilities in the frequency control voltage, the THz wave was also frequency-modulated (FM) at 333 Hz (which was much higher than the AM frequency). Through long operating time of the system with no service, Bidgoli et al. (2014) clearly proved that the THz radiation meets the optimal level of sensitivity requirement to not only overcome the hurdles facing IR measurement systems in an excellent way, but also to yield high-quality data with an acceptable temporal resolution. The replacement of slow conventional water measurement techniques with this new method allows rapid and stringent monitoring of gaseous steams from the biomass gasifiers, thereby providing better control over the whole process. The statistical analysis shows that with the electronic devices applied in the present work and the current design of the gas cell, the H_2O fractions in hot gaseous streams can be determined with an absolute precision close to 0.2% within a timescale of 1 min. This might be further improved on through optimization of the structural design of the gas cell and optimizing the THz transmitter/receiver system.

A more detailed analysis of the features of THz spectroscopy and possible fields of application one can find in Mittleman (2003), Schmuttenmaer (2004), Dexheimer (2007), Liu et al. (2007). Baxter and Guglietta (2011), Jepsen et al. (2011), Haddad et al. (2013), and Zouaghi et al. (2013).

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