

Spring 5-31-2013

Measurement of gas flow through porous structures using thz spectroscopy

Yihui Wang
New Jersey Institute of Technology

Follow this and additional works at: <https://digitalcommons.njit.edu/theses>



Part of the [Materials Science and Engineering Commons](#)

Recommended Citation

Wang, Yihui, "Measurement of gas flow through porous structures using thz spectroscopy" (2013).
Theses. 158.
<https://digitalcommons.njit.edu/theses/158>

This Thesis is brought to you for free and open access by the Electronic Theses and Dissertations at Digital Commons @ NJIT. It has been accepted for inclusion in Theses by an authorized administrator of Digital Commons @ NJIT. For more information, please contact digitalcommons@njit.edu.

Copyright Warning & Restrictions

The copyright law of the United States (Title 17, United States Code) governs the making of photocopies or other reproductions of copyrighted material.

Under certain conditions specified in the law, libraries and archives are authorized to furnish a photocopy or other reproduction. One of these specified conditions is that the photocopy or reproduction is not to be “used for any purpose other than private study, scholarship, or research.” If a user makes a request for, or later uses, a photocopy or reproduction for purposes in excess of “fair use” that user may be liable for copyright infringement,

This institution reserves the right to refuse to accept a copying order if, in its judgment, fulfillment of the order would involve violation of copyright law.

Please Note: The author retains the copyright while the New Jersey Institute of Technology reserves the right to distribute this thesis or dissertation

Printing note: If you do not wish to print this page, then select “Pages from: first page # to: last page #” on the print dialog screen

The Van Houten library has removed some of the personal information and all signatures from the approval page and biographical sketches of theses and dissertations in order to protect the identity of NJIT graduates and faculty.

ABSTRACT

MEASUREMENT OF GAS FLOW THROUGH POROUS STRUCTURES USING THZ SPECTROSCOPY

by

Yihui Wang

Comparing THz transmission through a sample with the transmission through free-space allows one to calculate the THz absorbance of a sample. Previous studies have focused on using THz absorbance to measure the diffusion of liquid through materials. In this study, the ability of THz spectroscopy to measure the flow of gas in a porous material, packaging foam, is investigated. Specifically, Terahertz spectroscopy from 0.5 to 0.7 THz is used to measure the THz absorption of 1,1-difluoroethane. Several experiments are performed to test the ability of THz spectroscopy to measure the flow of gas in a porous material: (1) The gas cell is empty (no foam) during the filling and purging of the gas cell. (2) Two layers of foam are put in the gas cell. The gas outlet at the bottom of the gas cell is left open. (3) Two layers of foam are placed in the gas cell. In this condition, the gas outlet at the bottom of the cell is left only partially open. The data shows that THz spectroscopy can be used to measure the flow of gas in opaque porous materials. The longer term goal of thesis research is to eventually use the THz properties of the gas to measure its diffusion through different porous products and correlate the gas diffusion with the material's structure.

**MEASUREMENT OF GAS FLOW THROUGH
POROUS STRUCTURES USING THZ SPECTROSCOPY**

**by
Yihui wang**

**A Thesis
Submitted to the Faculty of
New Jersey Institute of Technology
in Partial Fulfillment of the Requirements for the Degree of
Master of Science in Materials Science and Engineering
Interdisciplinary Program in Materials Science and Engineering**

May 2013

APPROVAL PAGE

**MEASUREMENT OF GAS FLOW THROUGH
POROUS STRUCTURES USING THZ SPECTROSCOPY**

Yihui Wang

Dr. John F. Federici, Thesis Advisor
Distinguished Professor of Physics, NJIT

Date

Dr. N.M. Ravindra, Committee Member
Chair of Physics, NJIT

Date

Dr. Robert B. Barat, Committee Member
Professor of Chemical Engineering, NJIT

Date

BIOGRAPHICAL SKETCH

Author: Yihui Wang

Degree: Master of Science

Date: May 2013

Undergraduate and Graduate Education:

- Master of Science in Materials Science and Engineering
New Jersey Institute of Technology, Newark, NJ, 2013
- Bachelor of Science in Physics
Chinese Culture University, Taipei, Taiwan, 2008

Major: Materials Science and Engineering

ACKNOWLEDGEMENT

The success of this study required the help of various individuals. Many thanks to my adviser, Dr. John Federici, who has given me the wisdom, support and knowledge for exploring things, and guidance at each step of my studies. My thanks goes to my committee member, Dr. Robert Barat, who reached out in every possible way to support this endeavor of providing invaluable suggestions and helping to set up experimental equipment. Also thanks to my committee member, Dr. N.M. Ravindra, who has supported me throughout my graduation program.

I am also grateful to the physics lab director, Sung M. Maeng, for his help with the equipment necessary for my experiments.

TABLE OF CONTENTS

| Chapter | | Page |
|---------|---|------|
| 1 | INTRODUCTION..... | 1 |
| 1.1 | Background..... | 1 |
| 1.2 | Objective..... | 3 |
| 2 | EXPERIMENTAL..... | 4 |
| 2.1 | Device..... | 4 |
| 2.2 | Gas Cell..... | 7 |
| 2.3 | Sample Data..... | 8 |
| 2.4 | Experimental Setup..... | 10 |
| 3 | RESULTS AND DISCUSSION..... | 13 |
| 3.1 | Analyzing Method..... | 13 |
| 3.2 | Experimental Results and Discussion..... | 13 |
| 3.2.1 | Absorbance of Gas Cell, Foam, and 1,1-difluoroethane..... | 13 |
| 3.2.2 | Determination of Foam's Resistance to Gas Flow..... | 16 |
| 3.2.3 | Determination of Foam's Resistance to Gas Flow-The Bottom Layer of Foam..... | 18 |
| 3.2.4 | The Top Layer of Foam Under Different Pressures..... | 20 |

| | | |
|---|----------------------------------|----|
| 4 | CONCLUSIONS AND FUTURE WORK..... | 23 |
| | REFERENCES..... | 25 |

TABLE OF FIGURES

| Figure | Page | |
|---------------|--|----|
| 1.1 | Electromagnetic spectrum including Terahertz range (Adapted from http://www.intechopen.com/books/recent-optical-and-photonics-technologies/terahertz-wave-parametric-sources , accessed February 20,2013)..... | 2 |
| 1.2 | The THz transmission of the cork sample at 10.9 hr, 21.9 hr, 33 hr, 44 hr, 55.6 hr, 78.2 hr, and 93.6 hr, respectively from left to right, top to bottom..... | 2 |
| 2.1 | T-Ray 2000 system..... | 6 |
| 2.2 | One commonly used design for photoconductive antenna (a) transmitter (b) receiver..... | 6 |
| 2.3 | The gas cell..... | 8 |
| 2.4 | Measured THz time-domain waveform (blue). The reference waveform is in green..... | 8 |
| 2.5 | Corresponding amplitude as a function of frequency after Fourier transforming the time-domain data..... | 10 |
| 2.6 | Corresponding phase amplitude as a function of frequency after Fourier transforming the time-domain data..... | 10 |
| 2.7 | The foam..... | 12 |
| 3.1 | Frequency dependent absorbance for the time-domain waveform | 14 |
| 3.2 | Frequency dependent absorbance for the time-domain waveform of 1,1-difluoroethane..... | 15 |
| 3.3 | The THz absorbance of the gas cell with pure nitrogen both with and without foam..... | 16 |
| 3.4 | The THz absorbance of the foam..... | 16 |

| | | |
|-----|---|----|
| 3.5 | Comparing absorbance of 1,1-difluoroethane in empty gas cell with that with top layer foam of the gas cell..... | 18 |
| 3.6 | Comparison of absorbance of 1,1-difluoroethane in empty gas cell with that of bottom layer foam, and the flushing out rate in the cell..... | 20 |
| 3.7 | Absorbance of top layer foam under different pressures..... | 22 |
| 3.8 | The top layer foam flushing out rate at 1.5 psi and 2.0 psi..... | 22 |

CHAPTER 1

INTRODUCTION

1.1 Background

Terahertz radiation consists of frequencies from 0.1 to 10 THz, occupying the region between microwaves at lower frequencies and infrared at higher frequencies, as shown in Figure 1.1. Since THz can pass through many materials that are impervious for visible light without considering ionizing radiation, it is applied in several fields, such as medical imaging, sensors, and non-destructive evaluation of examining material structures.[1] However, THz radiation is strongly absorbed by water vapor as well as liquid water. It is strongly reflected by metals.

Several research groups have shown the capability of using THz spectroscopy and imaging as a method to measure non-destructively the diffusion of liquids – in particular liquid water – through materials. Due to the relatively high absorbance of the diffusing liquid compared to the host material, one can measure the front of liquid as it diffuses as well as infer from those measurements the average as well as local diffusion coefficient of the material. One can use THz imaging of a material's internal structure to visualize the diffusion pathways. The cracks, voids, fissures, and imperfections in materials may present routes of rapid water diffusion. The penetration of a liquid with high permittivity into materials produces a contrast between dry and wet THz absorbance images. Figure 1.2 shows the THz transmission image of a 'dry' cork sample and the sample after injecting water. After introduction of water, the cork sample becomes highly absorbance. Since THz is good as seeing 'through' materials, it is a promising technique not only to detect the

presence of defects, cracks and imperfections, but also to directly monitor the flow or diffusion of gasses or liquids which can be strongly affected by the imperfections in a material.

While THz measurement techniques have been shown to be useful as a non-destructive evaluation tool for measurement of liquid diffusion in materials, there has been essentially no published work demonstrating the ability of THz measurement techniques to non-destructively measure the diffusion of gases through a porous structure.

[2]

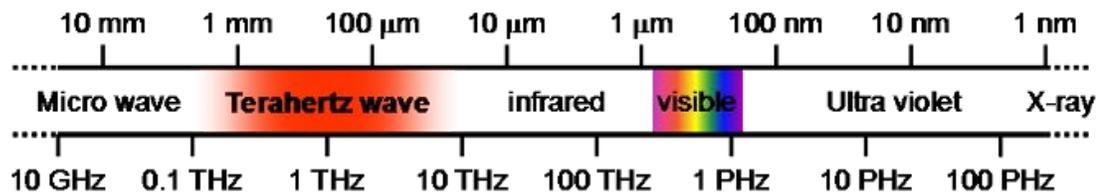


Figure 1.1 Electromagnetic spectrum including Terahertz range.

(Adapted from

<http://www.intechopen.com/books/recent-optical-and-photonics-technologies/terahertz-wave-parametric-sources>, accessed February 20, 2013)

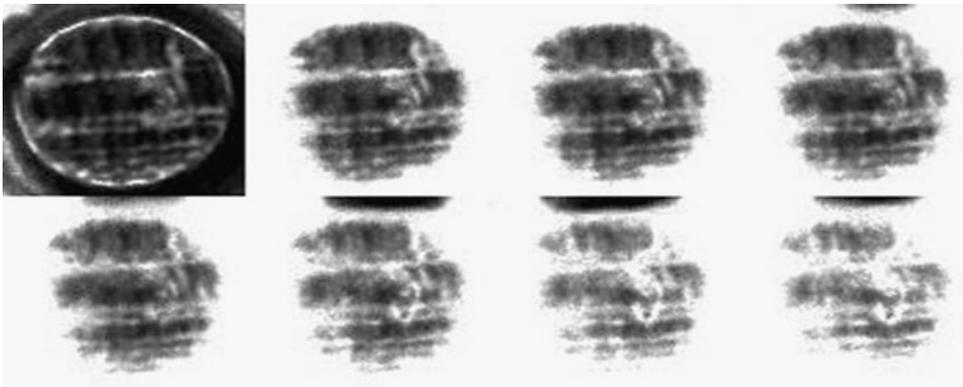


Figure 1.2 The THz transmission of the cork sample at 10.9 hr, 21.9 hr, 33 hr, 44 hr, 55.6 hr, 78.2 hr, and 93.6 hr, respectively from left to right, top to bottom. [3]

THz spectroscopy has been used as a tool to study a number of gases, such as carbon monoxide, nitric oxide, and methyl fluoride. There is interaction between the rotational energy states of gas molecules with THz radiation which creates absorption of the THz energy. The strength of interaction in THz region is more intense than interaction in microwave region, the interaction strength drops sharply towards the infrared region. Many gases obtain high absorption spectrums over a large bandwidth in the THz range. For example, 1,1-difluoroethane with the chemical formula $C_2H_4F_2$ is a colorless gas. It is commonly used as the main ingredient of gas dusters. It has a strong THz absorption between 0.5 THz and 0.7 THz.[1,4,5] In this thesis, the strong absorbance of 1,1-difluoroethane is used as the spectroscopic signature which enables one to measure the quantity of gas which permeates through a porous structure.

1.2 Objective

In this thesis, the goal is to use Terahertz spectra of a gas as a non-destructive evaluation tool to measure gas flow through a porous structure: specifically two layers of packing foams. In this thesis, the interaction between THz radiation and 1,1-difluoroethane is measured and the use of THz radiation for measuring THz absorbance of 1,1-difluoroethane through foam is emphasized. The experimental device and the setup are described in Chapter 2. In Chapter 3, the method of calculating THz absorbance and experimental results are detailed. The conclusions of this work are summarized in Chapter 4.

CHAPTER 2

EXPERIMENTAL

2.1 Device

There are several techniques to create radiation in the far-infrared or Terahertz region of the spectrum, such as free-electron lasers, gas lasers, and quantum cascade lasers. For this thesis research, a T-Ray 2000 spectroscopy system (Picometrix, Inc.) is used to measure the THz time-domain waveform. The T-Ray system is a THz time-domain spectrometer (THz-TDS) which generates and detects broad spectral pulses of THz radiation and simultaneously records the THz amplitude and phase information. This system includes a femtosecond lasers source, transmitter (Tx), a receiver (Rx), grating dispersion compensator, and a control box, as shown in Figure 2.1. [4, 6]

The Femtosecond Ti: sapphire laser source is applied for the generation and detection of THz radiation. The system produces optical pulses of ~100-fs duration, operating at a wavelength of ~800 nm. While other laser sources such as mode-locked fiber lasers can be used for THZ-TDS, Ti:Sapphire lasers are the most widespread femtosecond laser sources. These lasers provide excellent pulse-to-pulse and long-term stability.

The ultrafast optical lasers used with semiconductor photoconductive switches, also named Auston switches, is one technique for the generation and detection of THz pulses used in THz-TDS. This is the method employed by the T-Ray 2000 system. THz radiation is generated and detected through photoconduction arising from the flow of charge. Figure 2.2 (a) and (b) show the illustration of a photoconductive switch for transmitter and receiver. Photoconductive antennas are applied to transmitter and receiver

systems. These antennas consist of H-shaped metal lines deposited on semiconductor, GaAs. The spacing of the electrodes is from 5 μm to 100 μm . For the generation of THz pulses, these electrodes are applied on two metal lines with a voltage between 10V and 50V. Laser pulses are focused in between the metal lines at the gap. As a laser pulse illuminates the gap, charge carriers are injected and the antennas become conductive. The charge carriers are accelerated by the bias voltage field. The motion of carriers and their generation of freely propagating THz radiation can be described by Maxwell's equations. The time-varying electric current density gives rise to the emission of a time dependent electromagnetic field.

$$\nabla \times \mathbf{B} = \mu \mathbf{J} + \mu \varepsilon \frac{\partial \mathbf{E}}{\partial t} \rightarrow \nabla \times \mathbf{H} = \sigma \mathbf{E} + \frac{\partial \varepsilon \mathbf{E}}{\partial t} \quad (2.1)$$

where \mathbf{E} is electric field, \mathbf{B} is magnetic field, \mathbf{H} is magnetizing field, μ is the permeability of material, ε is the permittivity of material, and \mathbf{J} is current density. Any process which creates a time-dependent change in the material properties μ or ε , can act as a current source that results in emission of THz radiation. A semiconductor device causes rapid changes to the material properties, which are represented by σ , ε , and μ . Together, with an electric bias on the two metal lines, a THz wave is generated. [7]

For receivers, there is no bias voltage applied to the electrodes. The bias is replaced by a sensitive ammeter or transimpedance amplifier so the tiny current can be detected which is proportional to the THz electric field. An incoming THz electric field will cause

the generated carriers to be displaced leading to a current flow during the lifetime of the photo-carriers.[4, 8, 9]

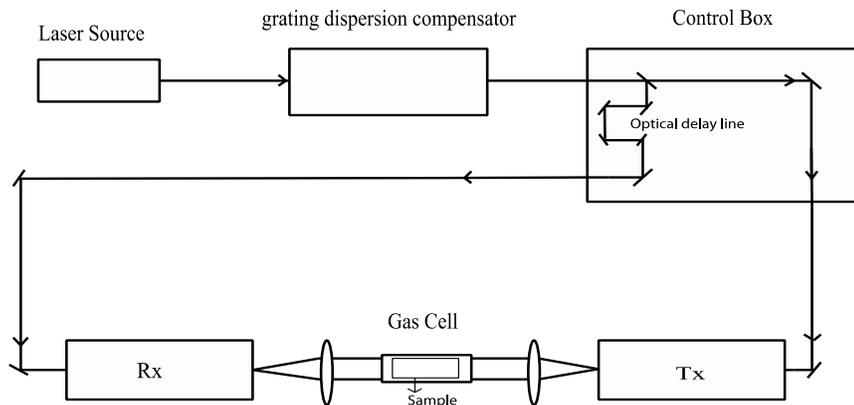


Figure 2.1 T-Ray 2000 system

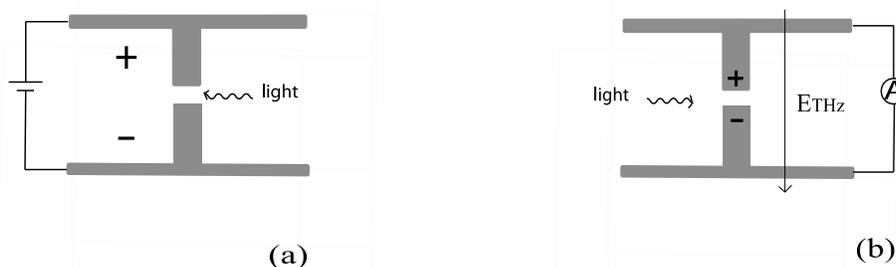


Figure 2.2 One commonly used design for photoconductive antenna (a) transmitter (b) receiver.

Experimentally the laser pulse is split into two arms, one for the THz emitter and one for the detector. In order to synchronize the timing arrival of the incoming THz radiation from the THz transmitter with the laser pulse which activates the THz receiver, a mechanical delay line is used to adjust the optical path length for the laser pulse before it interacts with the THz detector. When the laser pulse interacts with the THz detector, the detector is gated or ‘turned on’ for a brief period of time corresponding to the time duration

of the laser pulse. During this brief period of time ($\sim 100\text{fs}$), essentially only one point in the THz pulse is measured. In order to move the detection gate to another time point in the THz pulse, the delay line needs to be adjusted in length or equivalently in time delay. The control box is used to control the delay time. By repeatedly changing the time delay, the THz-TDS system maps out the measured THz waveform as a function of time.

Phase and amplitude information is extracted from the THz time-domain waveform by applying a Fourier Transform to convert time domain data to the frequency domain. As will be shown in Section 2.3, the Fourier Transform yields the spectrally dependent amplitude and phase of the THz radiation. From the amplitude and phase, and knowing the length of a sample, the absorption coefficient and real refractive index of a sample can be calculated.

2.2 Gas Cell

The body of the gas cell is made of plastic. The size is 10cm length by 10cm width by 22cm height. Between the plastic and metal, a rubber gasket is inserted. In this experiment, the body of this gas cell is enclosed by a piece of metal at each end. The size of each piece of metal is 14cm length by 14cm width by and 0.5 cm height. Gas fittings are attached to the metal end pieces to allow the gas sample to be delivered to the cell using standard flexible hose connections. Two layers of foam are enclosed in this plastic chamber; the size of the foam is 12cm length by 12cm width by 7cm height. Figure 2.3 shows the gas cell.

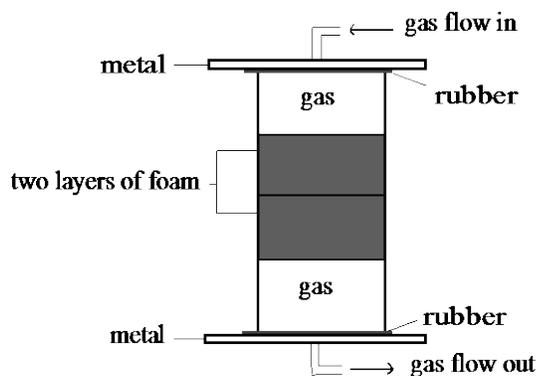


Figure 2.3 The gas cell.

2.3 Sample Data

The result of a typical measurement of THz time-domain waveforms is shown Figure 2.4, which is recorded as electric field as a function of time delay. When a sample is inserted between the detector and transmitter, the pulse is delayed due to the refractive index of the sample. The THz phase and amplitude is normalized to the phase and amplitude of the reference.

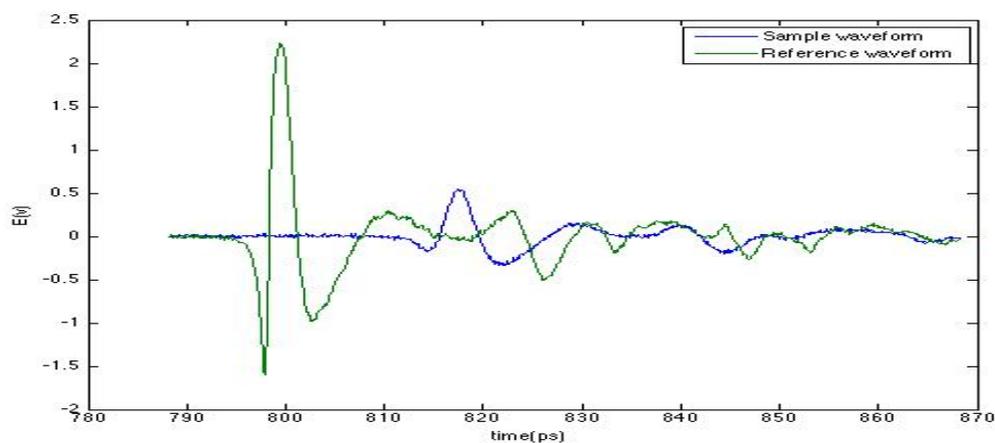


Figure 2.4 Measured THz time-domain waveform (blue). The reference waveform is in green.

A Fourier transform is used to convert the time domain data into the frequency domain. In frequency domain data, an amplitude and phase change at every frequency can be obtained, as shown in Figure 2.5 and Figure 2.6. THz amplitude approaches the noise limit of the THz system when the THz amplitude approaches a roughly constant average value. For reference spectra of Figure 2.5, this occurs at approximately 2.1 THz. For sample data, all data above a frequency of ~ 0.5 THz is in the noise. [10]

In order to extract the absorbance and real refractive index from the THz spectral data, the sample THz phase and amplitude is normalized to the phase and amplitude of the reference waveform. The refractive index can be obtained by calculating the difference between sample phase and reference phase divided by the THz frequency.

$$n_r = 1 + \frac{\phi_s - \phi_r}{2\pi} \left(\frac{c}{\nu L} \right) \quad (2.2)$$

where n_r is refractive index, ϕ_s and ϕ_r are sample phase and reference phase respectively, c is speed of light, ν is frequency, L is length of sample. From Figure 2.6, before approaching the noise, the phase shift is linear. The absorbance of the sample can be calculated simply using the formula $A = \ln \frac{|E_r|}{|E_s|}$.

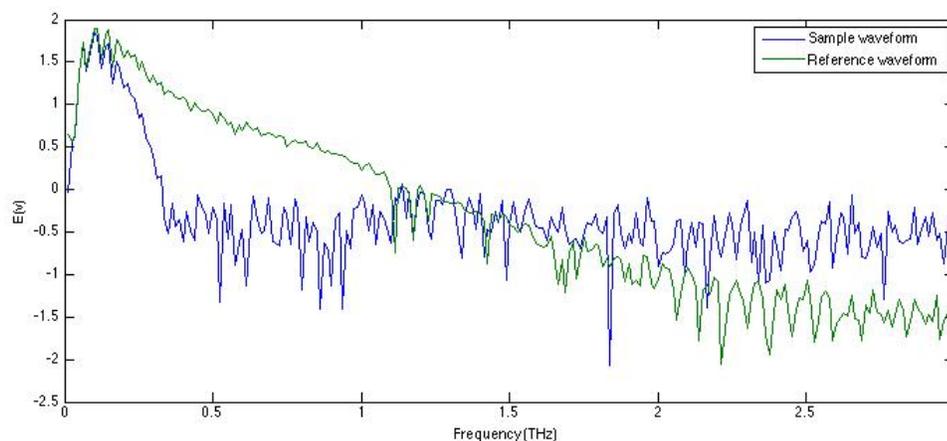


Figure 2.5 Corresponding amplitude as a function of frequency after Fourier transforming the time-domain data.

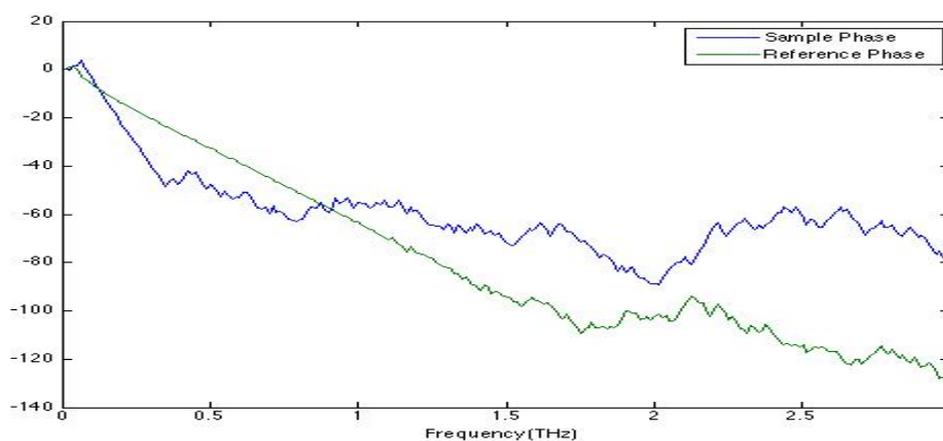


Figure 2.6 Corresponding phase amplitude as a function of frequency after Fourier transforming the time-domain data.

2.4 Experimental Setup

In order to determine the appropriate frequency range for analyzing the flow of gas through a porous medium, THz absorbance of 1,1-difluoroethane is measured in an otherwise empty cell (no foam). Nitrogen is used to flush out any residual 1,1-difluoroethane in the gas cell. The THz waveform is measured through the nitrogen filled cell (1 atm). One

thousand waveforms are averaged for this experiment. Subsequent to this background measurement, the gas cell is filled with 1,1-difluoroethane (1 atm). Using these measurements, the THz transmission through 1,1-difluoroethane is compared with the transmission through free-space. As described in the next chapter, the transmission data with 1,1-difluoroethane and with nitrogen are compared to determine the absorbance spectra of 1,1-difluoroethane.

There are several cycles to characterizing the flow of gas through the porous foam. In each cycle, 1,1-difluoroethane is injected into the cell by opening a valve. THz absorbance of 1,1-difluoroethane on the top layer of foam is recorded as a function of time. For these time dependent measurements, 10 THz time-domain waveforms are measured and averaged every 100ms. Gas is flowed into the chamber until a roughly constant value of attenuation is attained. Nitrogen is then injected into the cell to flush out 1,1-difluoroethane. Again, a time sequence of THz spectra is acquired. Using these measurements, the rate filling and flushing-out of 1,1-difluoroethane through the porous foam is studied. Several cycles of the filling and flushing are repeated under different pressure conditions. Measurements were performed with the gas cell empty or with two layers of foam in the gas cell. The foam is a standard plastic foam material which is used as cushioning packing material.

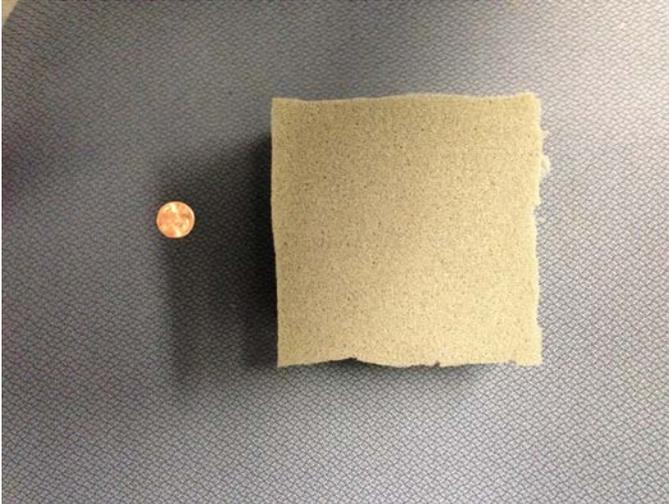


Figure 2.7 The foam.

In order to test the ability of THz spectroscopy to measure the flow of gas in a porous material, several experiments are performed:

1. Experiment 1: The gas cell is empty (no foam layers) during the filling and purging of the gas cell.
2. Experiment 2: Two layers of foam are put in the gas cell. In this condition, THz beams are set to go through the top layer and the bottom layer of foam. The gas outlet at the bottom of the gas cell is left open. In this configuration the intent was for the foam to restrict the flow of gas through the gas cell.
3. Experiment 3: Two layers of foam are placed in the gas cell. In this condition, THz beams are set to go through the top layer of foam. The gas outlet at the bottom of the cell is left only partially open. In this configuration the intent was for the partially open exit valve to restrict the flow of gas through the gas cell.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Analyzing Method

There are many methods for analyzing the THz waveform. In this study, the THz frequency dependent optical constants of the THz pulse through the gas cell are investigated. The THz phase and amplitude is normalized to the phase and amplitude of the reference waveform. The absorbance (A) of the sample are calculated by comparing THz transmission through the sample with that through free-space, by using the equations:

$$A = \ln \frac{|E_r(\nu)|}{|E_s(\nu)|} \quad (3.1)$$

where $|E_r(\nu)|$ and $|E_s(\nu)|$ are the magnitudes of the reference and sample THz electric fields as calculated by the Fourier transform, respectively. [3]

3.2 EXPERIMENTAL RESULTS AND DISCUSSION

3.2.1 Absorbance of Gas Cell, Foam, and 1,1-difluoroethane

For the spectroscopic absorbance measurement of 1,1-difluoroethane, the THz waveform is measured both with 1 atm of nitrogen (reference) and 1 atm of 1,1-difluoroethane (sample). The absorbance of the 1,1-difluoroethane gas can be calculated from Eq. 3.1 and the data of Figure 2.5. The appropriate THz bandwidth for subsequent data processing is

determined by measuring the spectral width of the 1,1-difluoroethane gas absorbance spectra. THz frequency domain data is shown in Figure 3.1. A thousand THz waveforms are averaged for both the reference and sample spectra. The sample waveform drops between 0.5 and 0.7 THz, which indicates the peaks of absorbance is located in this spectral interval. Using Eq. 3.1, the THz absorbance of 1,1-difluoroethane in empty gas cell is shown in Figure 3.2. It corresponds with Figure 3.1. There is no signal below 0.05 THz. Absorbance approaches the noise at approximately 1 THz. This frequency interval of 0.5-0.7 THz is used in the remainder of the analysis to determine average THz absorbance relative to the reference spectrum. The average absorbance in the 0.5-0.7 THz band should be proportional to the average concentration of 1,1-difluoroethane gas in the gas cell structure.

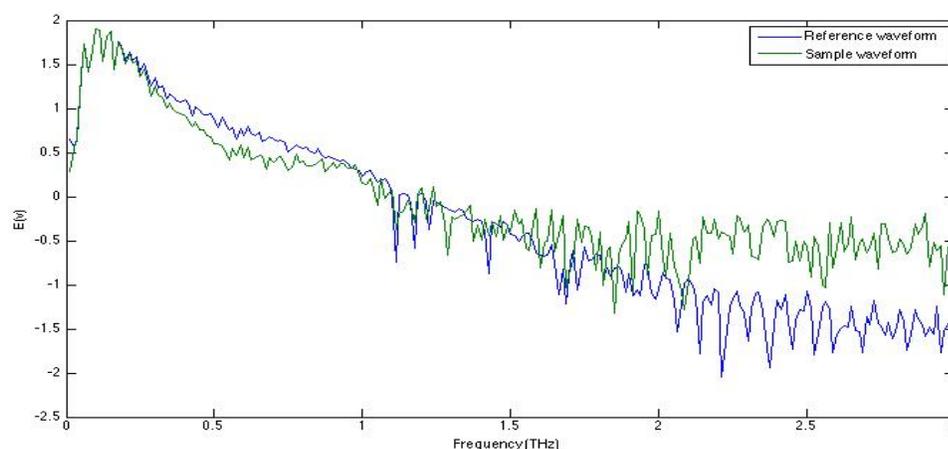


Figure 3.1 Frequency dependent absorbance for the time-domain waveform.

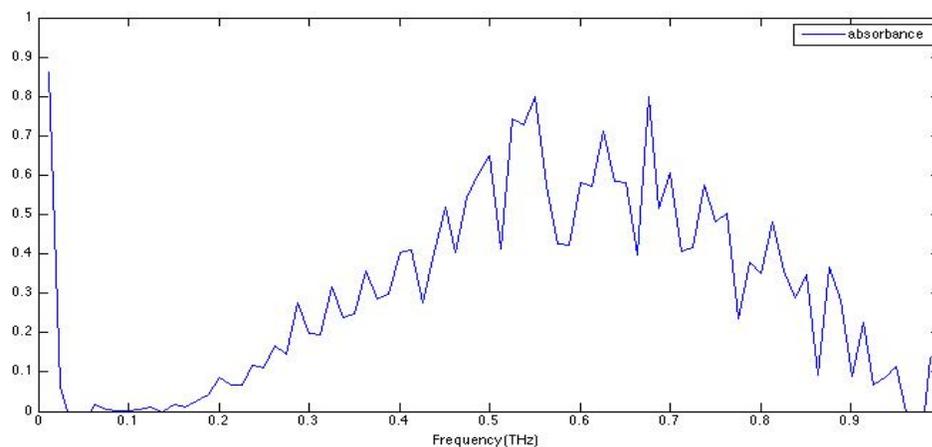


Figure 3.2 Frequency dependent absorbance for the time-domain waveform of 1,1-difluoroethane.

Figure 3.3 shows the THz absorbance of the gas cell with foam (black) and with no foam (green). There is no signal below 0.05 THz. The black line approaches the noise at approximately 0.7 THz. To compare the THz absorbance of the gas cell and foam with the THz absorbance of the plastic gas cell, the THz absorbance of the gas cell is relatively low. Figure 3.4 shows the THz absorbance of foam only. There is no signal below 0.06 THz. Absorbance approaches the noise at approximately 0.8 THz. This absorbance waveform suggests that the foam is transparent enough to detect THz transmission. On the other hand, the THz absorbance of the foam is high enough, which allows us to use the THz transmission of the foam and the gas cell as reference waveform to determine average THz absorbance relative to the sample spectrum.

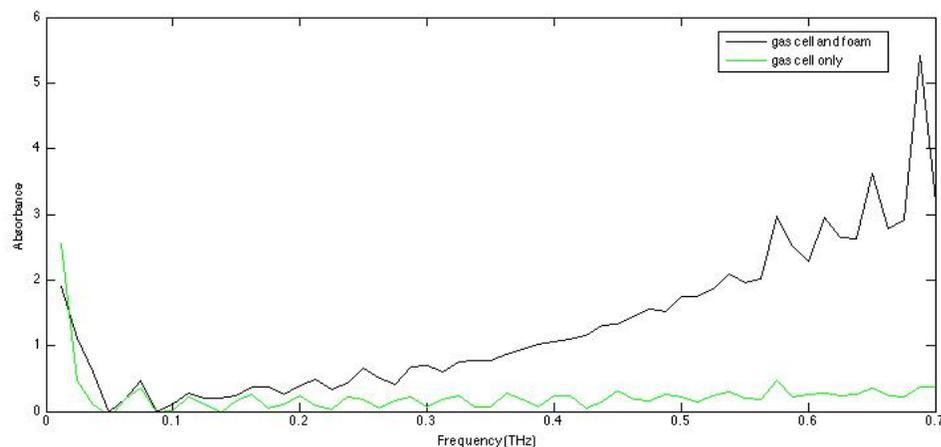


Figure 3.3 The THz absorbance of the gas cell with pure nitrogen both with and without foam.

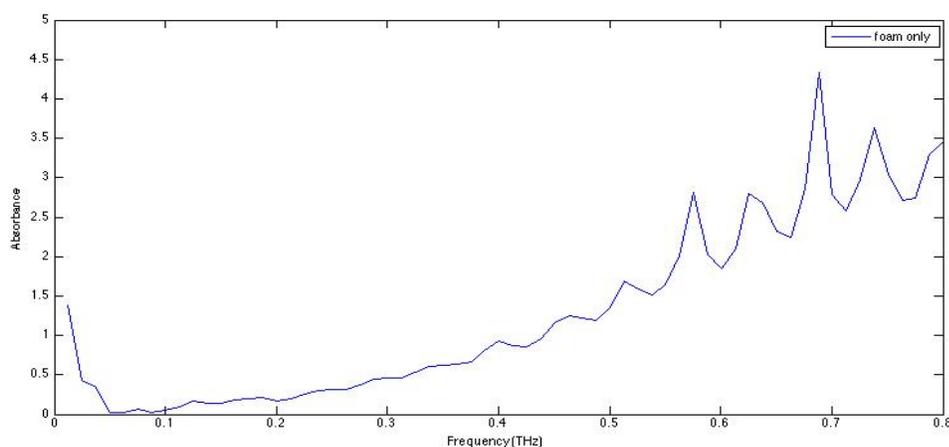


Figure 3.4 The THz absorbance of the foam.

3.2.2 Determination of Foam's Resistance to Gas Flow

By comparing the results of Experiment 1 to Experiment 2, one can determine whether or not the foam contributes a measurable resistance to the flow of gas in the cell. Essentially, we measure the time-dependent absorbance in the gas cell with the exit valve fully open. Measurements are made with the foam in the gas cell and with the foam removed. In this experiment, when the foam is present, THz beams is aligned to penetrate through the top

layer foam. Figure 3.5 shows the comparison of THz absorbance of 1,1-difluoroethane in the empty gas cell compared to that of the top layer of foam in the gas cell. The absorbance at different time is form by calculating the average absorbance from 0.5 THz to 0.7 THz.

The blue line indicates THz absorbance of 1,1-difluoroethane in the gas cell without foam; the red line shows the THz absorbance of 1,1-difluoroethane of the top layer of foam. Some of the features of the data are consistent with expectations while others are not consistent. The blue line reaches the saturated state (the gas cell is filled with 1,1-difluoroethane) at the value of absorbance around 2.7. The red line reaches the saturated state at the value of absorbance around 1.4. This is expected since when the foam is present, a smaller volume of gas is present in the gas cell since the foam itself is occupying some of the space. Thus, when the saturated state is reached, less gas is present in the chamber with foam than without foam.

On the contrary, the time-dependence of the data exhibits some inconsistencies. The blue and the red line start at zero and increase as time increases until a saturated state is reached. The blue line reaches the saturated state around the eighteenth second; the red line reaches the saturated sate around the twentieth second. Ideally, it should take shorter time to reach saturation for 1,1-difluoroethane flowing through the empty cell. This experimental result strongly suggests that the foam does not restrict the flow of gas. The gas flow is restricted mostly by the valve because the foam is too porous.

The green line shows the flushing-out process of the top layer of foam. The value of the curve decreases as time increases. This line goes down at the value of absorbance is near 1.4. It corresponds to the saturated state of injected 1,1-difluoroethane. The absorbance is below zero after the saturated state is reached. This is probably due to some

residual 1,1-difluoroethane in the cell when the reference scan was acquired. The small amount of 1,1-difluoroethane in the reference scan (supposedly after all 1,1-difluoroethane had been removed) could account for a lower absorbance value after prolonged flushing with nitrogen.

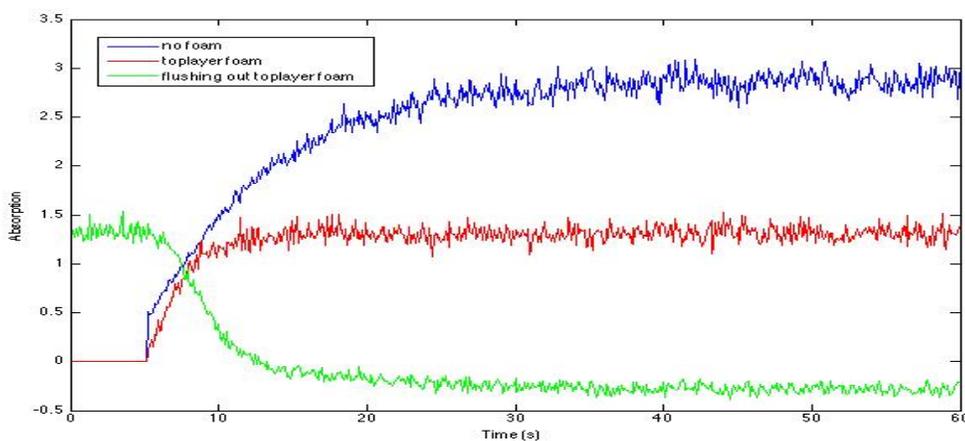


Figure 3.5 Comparing absorbance of 1,1-difluoroethane in empty gas cell with that with top layer foam of the gas cell.

3.2.3 Determination of Foam's Resistance to Gas Flow -The Bottom Layer of Foam

To ensure that our conclusion that the foam was not inhibiting the flow of gas in the cell, Experiment 1 and 2 were repeated except with the THz beam propagating through the bottom most layer of foam. By propagating through the bottom most layer, it would be more certain that any gaps between the foam and the plastic walls were plugged forcing the gas to pass through the porous foam structure.

Figure 3.6 is THz absorbance and the flushing out rate for the bottom layer of foam. The blue line indicates the THz absorbance of 1,1-difluoroethane in the gas cell without foam; the red line shows the THz absorbance of 1,1-difluoroethane of the bottom layer of foam. These two lines start at zero absorbance at 6.6 second. The values of absorbance

increase as the time increases until the saturated state is reached. The blue line reaches the saturated state near the thirtieth second; the red line reaches the saturated state around the eightieth second. Ideally, it takes shorter time to reach saturation for 1,1-difluoroethane goes through the empty cell. This experimental result is consistent with the results for Experiment 1 and 2 through the top layer of foam: the foam does not restrict flow of gas. The gas flow restricted mostly by the exit valve.

Before the red line starts to increase, the line drops a little from time at zero second to 6.6 second. The reason to cause this situation is that in the previous test, the 1,1-difluoroethane in the cell with the top layer of foam is flushed-out by nitrogen. Even though 1,1-difluoroethane is flushed-out completely in the top layer of foam, there is still some gas that is residual in the bottom layer of foam and nitrogen gas is still in the top layer of foam. As 1,1-difluoroethane is injected into the gas cell, it pushes the residual nitrogen through to the bottom layer of foam and flushes-out the residual 1,1-difluoroethane in this layer, which causes the red line drops from the absorbance value around 0.4 to zero before 6.6 second.

The blue line reaches the saturated state at the value of absorbance around 3. The red line reaches the saturated state at the value of absorbance around 1.8. This is constant with a smaller volume of gas present with the foam since the foam itself occupies a fraction of the volume which is probed by the THz radiation.

The green line shows the flushing-out process of the top layer of foam. The line goes down as time increases. The line drops at absorbance value around 1.8, which corresponds to the saturated state of injected 1,1-difluoroethane. The absorbance

approaches zero when it is reaching the saturated state. This verifies that the 1,1-difluoroethane is flushed-out from this layer of foam.

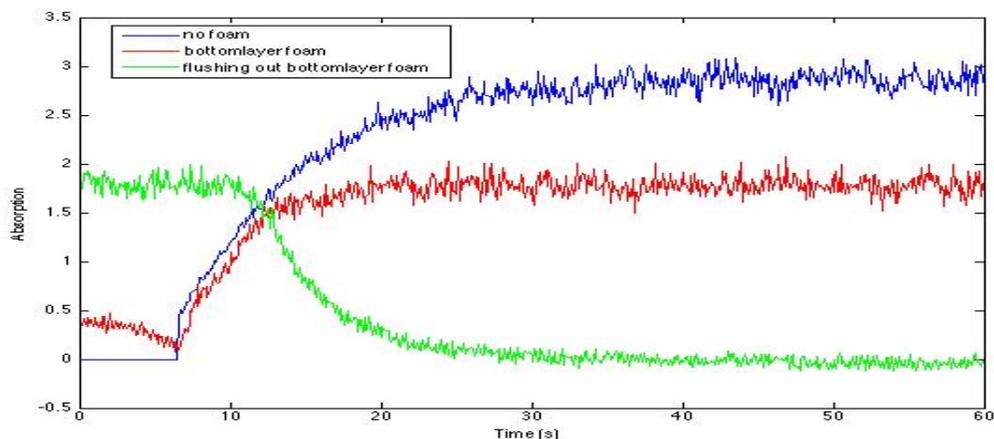


Figure 3.6 Comparison of absorbance of 1,1-difluoroethane in empty gas cell with that of bottom layer foam, and the flushing out rate in the cell.

3.2.4 The Top Layer of Foam Under Different Pressures

As discussed in the previous section, there was essentially no difference in the rate of increase of 1,1-difluoroethane gas in the gas cell whether or not the foam is present. This result suggests that the foam has little effect on the gas flow through the cell since it is too porous. The flow of gas through the gas cell is predominately determined by the diameter of the entrance and exit hosing into the cell. In order to more controllably adjust the flow of gas into the cell, the exit hose was partially closed so that the restricted size of the opening limited the gas flow through the cell. With this restriction, the gas pressure inside of the gas cell could be maintained at a roughly constant value during the filling and flushing procedure.

With the foam in place, and the gas outlet from the gas cell partially closed, the THz absorbance of 1,1-difluoroethane under filling pressures of 0.75 psi, 1.6 psi, 1.9 psi and 2.5

psi is recorded as a function of time. The flushing rates of 1,1-difluoroethane gas with nitrogen at 1.5 psi and 2.0 psi are recorded as a function of time.

In this experiment, THz beams are set to go through the top layer of foam. The THz absorbance of the top layer of foam as a function of time under different pressures is shown in Figure 3.7. The black line shows the THz absorbance for 0.75 psi; the green line is the THz absorbance for 1.6 psi. The red and blue lines are the THz absorbance processes for 1.9 psi and 2.25 psi, respectively. The data has been shifted in time so that equivalently the gas starts to flow into the cell at the same time. These four lines all start near zero absorbance. The absorbance starts to increase around 6.5 second. The values of absorbance increase as time increases. The black line reaches the saturated state around 50s; the blue and green lines reach the saturated state around 20s; the red line reaches the saturated state near 15s. Theoretically, one would expect it to take longer time to reach the saturated state under lower pressure. The test result indicates that it takes longer time to reach the saturated state under 0.75 psi. However, there is not much difference at higher pressures, which is attributed to the fact that the foam does not restrict the flow of gas. The flow of gas is restricted primarily by the restricted size of the exit valve.

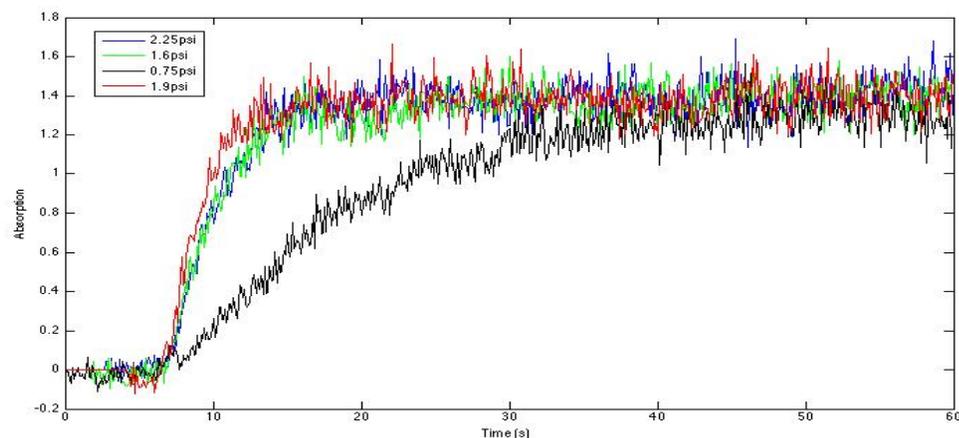


Figure 3.7 Absorbance of top layer foam under different pressures.

Figure 3.8 shows the flushing-out rates at 1.5 psi (green) and 2.0 psi (pink). The values of absorbance drop as time increases. The data has been shifted in time so that equivalently the gas starts to flow into the cell at the same time. These two lines start to drop at the fifteenth second. The pink line approaches the saturated state near the thirtieth second; the green line approaches the saturated state near the fortieth second. Theoretically, it takes a shorter time to flush-out 1,1-difluoroethane under higher pressure. This experimental result matches what is expected.

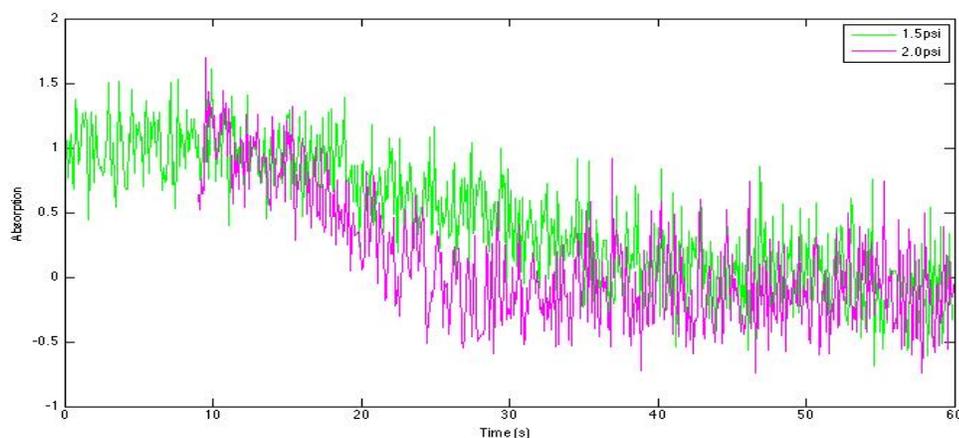


Figure 3.8 The top layer foam flushing out rate at 1.5 psi and 2.0 psi.

CHAPTER 4

CONCLUSIONS AND FUTURE WORK

In this thesis, THz spectroscopy is applied to non-destructively measure the diffusion of gases through a porous structure. Using a time-domain THz spectroscopy system, THz transmission in the 0.5-0.7 THz range is used to obtain the average THz absorbance relative to the reference spectrum. The flow of gases through two layers of foam is measured. During the filling process, the value of absorbance of both two layers of foam increases as time increases. When the saturated state is reached, less gas is present in the gas cell with foam than without foam since the foam occupies some of the space of the gas cell. However, since the foam is too porous, the foam does not restrict the flow of gas. In order to more controllably adjust the flow of gas into the cell, the exit hose was partially closed. The THz absorbance of 1,1-difluoroethane under filling pressures of 0.75 psi, 1.6 psi, 1.9 psi and 2.5 psi and flushing rates of 1,1-difluoroethane gas with nitrogen at 1.5 psi and 2.0 psi are recorded as a function of time. For the study of filling pressure of 0.75 psi, 1.6 psi, 1.9 psi and 2.5 psi, it takes longer time to reach saturation at 0.75 psi. However, there is no much difference at higher pressures since the foam does not restrict the flow of gas. This result indicates that the flow of gas is restricted primarily by the restricted size of the exit valve.

The gas diffusion through another kind of foam (a sprayed foam used for thermal insulation around pipes and windows) was also tested. However, the porosity of the spray foam is too low. The foam (about 10cm thick) effectively blocked the gas passing through the gas cell chamber. The flow or diffusion of gas through the foam was not measureable during a roughly 5 minute time interval of data acquisition.

Based on the analysis of the THz data this far, it appears that THz spectroscopy can be used to measure the flow of gas in opaque porous materials. Time dependence of gas flow can be obtained by THz spectroscopy. However, the work in this thesis does not complete the task of measuring the diffusion of gas in porous materials. In the next step of this work, a foam material should be chosen which resists the flow of gas (i.e. more resistive than the packing foam used in the thesis) but does not severely block the flow of gas through the sample (i.e. more permeable to gas flow than the spray foam). Ideally, the gas diffusion through a foam with some permeability to gas flow could be measured. After measuring the filling and flushing-out process, one can explore the relationship between the different foam products and the measured gas diffusion in these porous and permeable materials. If one were able to correlate the THz spectra with gas diffusion for different foam products, THz spectroscopy could be used as a non-invasive and non-destructive evaluation for quality control of foam products.

REFERENCES

1. D.M.Mittleman, R.H.Jacobsen., R. Neelamani, R.G. Baraniuk, M.C. Nuss, *Gas Sensing Using Terahertz Time-Domain Spectroscopy*. Applied Physics 1998. **B 67**: p. 379-390.
2. John F. Federici, *Review of Moisture and Liquid Detection and Mapping Using Terahertz Imaging*. Journal of Infrared, Millimeter, and Terahertz Waves, 2012. **33**(2): p. 97-126.
3. Anthony J. Teti, David E. Rodriguze, John F. Federici, *Non-Destructive Measurement of Water Diffusion in Natural Cork Enclosures Using Terahertz Spectroscopy and Imaging*. Journal of Infrared, Millimeter, and Terahertz Waves, 2011. **32**(4): p. 513-527.
4. D.Mittleman, *Sensing with Terahertz Radiation*. Springer (Berlin, Germany) 2003.
5. Mira Naftaly, Richard A. Dudley, *Calibration of Terahertz Spectrometers*. The International Society for Optical Engineering, 2011. **8195**: p. 8195N/1-8195/N-4.
6. *T-Ray 2000 Research Application Development*, Retrieved April 5, 2013; Available from: http://www.picometrix.com/pico_products/terahertz_research.asp.
7. R. A. Cheville, *Terahertz Spectroscopy Principles and Applications*. CRC Press (Boca Raton, FL, USA) 2008.
8. H. P. Merbold, *Terahertz Time-Domain Spectroscopy of Aqueous Systems in Reflection Geometry and Construction of Polarisation-Sensitive Photoconductive Terahertz Antennas*, Retrieved February 26, 2013; Available from: http://frhewww.physik.uni-freiburg.de/terahertz/frames/pdf/Merbold_Diploma.pdf.
9. M.Theuer, F.Ellrich, D.Molter, R.Beigang, *Handbook of Terahertz Technology for Imaging, Sensing, and Communications*: Woodhead Publishing (Philadelphia, PA, USA) 2013.
10. Yew Li Hor, John F. Federici, Robert L. Wample, *Nondestructive Evaluation of Cork Enclosures Using Terahertz/Millimeter Wave Spectroscopy and Imaging*. Applied Optics, 2008. **47**(1): p. 72-78.