Materials Research Express

PAPER

Using microfluidic technology to measure the terahertz absorption properties of phase-changing thermoregulated emulsions

Jing-Yi-Ran Jin¹, Bo Peng¹,²,³,⁴, Qing-Hao Meng¹,²,³,⁴, Si-Yu Qian¹,²,³,⁴, Bo Su¹,²,³,⁴,*, Hai-Lin Cui¹,²,³,⁴ and Cun-Lin Zhang¹,²,³,⁴

¹ Department of Physics, Capital Normal University, Beijing 100048, People’s Republic of China
² Key Laboratory of Terahertz Optoelectronics, Ministry of Education, Beijing 100048, People’s Republic of China
³ Beijing Key Laboratory for Terahertz Spectroscopy and Imaging, Beijing 100048, People’s Republic of China
⁴ Beijing Advanced Innovation Centre for Imaging Theory and Technology, Beijing 100048, People’s Republic of China
* Author to whom any correspondence should be addressed.

E-mail: subo75@cnu.edu.cn

Keywords: THz, microfluidic technology; phase-changing thermoregulated emulsions, paraffin molecules

Abstract

Phase-changing materials (PCMs) are widely used thermal-energy-storage substances that utilize the absorption and emission of heat during the solid–liquid–solid phase change of a substance to store and release thermal energy, which is closely related to their structural properties. This paper combines THz technology with microfluidic technology to measure the terahertz absorption properties of phase-changing thermoregulated emulsions and the results show that: with the increase of the applied magnetic field strength, more molecules are magnetized and arranged with orientation, and the transmission intensity of THz increases; with the increase of the applied electric field strength, the bond length of covalent bonds becomes larger, and the transmission intensity of THz increases; during the cooling process, the free energy of molecules decreases, and the transmission intensity of THz increases. The results provide technical and data support for the in-depth study of phase-changing thermoregulated emulsions, which is important for the fine study and improvement of properties in different environments in aerospace and coating fields.

1. Introduction

Terahertz (THz) waves are electromagnetic waves with frequencies in the range from 0.1 to 10 THz (wavelengths of 3000 to 30 μm) [1]. In recent years, with the boom in THz research and development, THz technology and its applications have started to find applications in many fields. In informatics, THz wireless local area networks and cellular networks can provide both ultra-high data-transmission rates of several terabits per second and high-speed wireless data distribution [2]. In medicine, THz polarization sensors can detect the proliferation of anti-tumor cells [3]. In life sciences, THz technology combined with other techniques can determine the complex dielectric constants of cellular monolayers, which allows us to determine the intracellular-dynamics properties of water molecules at the picosecond timescale [4]. In food studies, Seung et al performed spectroscopic detection of melamine mixtures in different matrices in the frequency range 0.1–3 THz, and they showed that the absorption peaks of melamine are independent of the matrix so that THz waves can be used to detect melamine in food [5]. THz time-domain spectroscopy (THz-TDS) is a new spectroscopic detection method with high penetration, low energy, high resolution, and a rich ‘fingerprint spectrum’ that can be used to characterize many different substances. Because different substances exhibit different absorption characteristics in the THz band, THz observations provide simple, fast, and effective means for detecting and characterizing different substances. For example, since different hydrates of metal sulfates exhibit different THz absorption properties, Ruggiero et al. successfully distinguished and characterized five hydrated forms of ferrous sulfate using the THz-TDS system and density functional theory (DFT) [6]. After obtaining the frequency-domain spectra of the samples using a THz spectrometer and Fourier transform, the DFT was used to analyze the
structures of anhydrous FeSO₄ and Fe₂(SO₄)₃·2H₂O, and to provide the frequency and intensity of the specified mode types of FeSO₄·4H₂O crystals. An unusual low-frequency feature in FeSO₄·7H₂O was also found. It was thought to be caused by the coupling of isolated electron charges with long range optical phonons. Bernd M. Fischer et al. used a THz-TDS system to measure the time-domain spectra of α-D-glucose and β-D-glucose, achieving the distinction between molecules with small structural differences [7]. Oppenheim et al. measured the characteristic absorption spectra of 1,2-benzencarbonitrile and 1,3-benzencarbonitrile at 77 K using a waveguide THz-TDS system, and they succeeded in discriminating between these two isomers because of the significant differences in their THz absorption spectra [8].

In recent years, microfluidics has been widely used in medical, chemical, and chromatographic research because of its many advantages, including miniaturization, low consumption of reagents, fast reaction speed, and designable flow-channel structure. Van Esch’s group found that crescent-shaped microgels containing glucose oxidase (GOX) can be carried through a microfluidic device, and because these microgels have high affinity and good selectivity for lung cancer cells, the investigators were able to achieve selective killing of 90% of the cancer cells that were captured in the microgel cavity without killing external cells [9]. Kingkan et al. prepared a paper-based microfluidic electrochemical-detection platform based on screen-printed graphene electrodes for the simultaneous detection of tin and lead, with detection limits of 0.26 and 0.44 ng ml⁻¹ for tin and lead, respectively [10]. Wang et al. showed that using microfluidics to make a sandwich-type chip not only reduced the absorption of THz waves by water, but also it is simple to fabricate, takes less time to use, and does not have leakage problems when heated [11].

Meng et al. [12] combined THz with microfluidic technology and studied the influence of different electric field action times on hydrogen bonds in solution, indicating that the process of ionic hydration affects hydrogen bonds between molecules in water. Zhang et al. [13] designed and manufactured a microfluidic chip with thousands of channels, combined with a coherent optical hybrid THz spectrometer, to probe the THz absorption characteristics of 60-nucleotide microcystins mixture dissolved in TE buffer at concentrations of 0.92 and 0.23 μg μl⁻¹. A distinctive feature was found recurring around 830 GHz. Rasha et al. [14] integrated a microfluidic platform with a THz-TDS system and developed a semi-classical computational model to simulate THz radiation emitted from a Gallium arsenic photoconducatance THz emitter. They found that many microfluidic-based THz-TDS systems had maximum measurable absorption coefficients with varying dynamic range values and sample thicknesses. And the corresponding maximum measurable frequency of THz-TDS system based on microfluidic is found. These studies show that THz technology and microfluidic technology have a very wide application prospect.

Phase-changing thermoregulated (PCT) emulsions are aqueous dispersions that contain microcapsules of a phase-changing material with a solid content in the range 34%–36%; they are usually used in solution spinning, coating, and other processes. Based on the THz-TDS technique, after obtaining the time-domain data of the sample, the fast Fourier transform can obtain the frequency domain information of the sample, and further theoretical calculations can obtain the refractive index and absorption coefficient of the sample, so as to analyze the intermolecular interaction forces and material structure. The various PCT emulsions used in life are usually present in aqueous solutions, whereas water will have strong absorption of THz waves. Microfluidics can then reduce the THz absorption of water by shortening the distance between THz and the sample and controlling its scale in a certain dimension in the micron or even nanometer range. The Cyclic Olefin Copolymer (COC) used to make the microfluidic chip also has a high transmission rate in the THz band [15]. Therefore, this study combines THz technology and microfluidics to obtain the PCT emulsions whose structure changes under different magnetic fields, electric fields and temperatures, resulting in different THz absorption. The results of this study provide data support for further in-depth research on the structure, provide new ideas and methods for the study of PCT emulsions, and are also important for the study of the performance of coatings made with PCT emulsions under different environments and how to improve them.

2. Experimental systems

2.1. Experimental optical path system
The THz-TDS system used in the experiment is shown in figure 1. The laser output from the fiber femtosecond laser (center wavelength 1550 nm, pulse width 75 fs, output power 130 mW, pulse repetition frequency 100 MHz) is split into two beams by a polarization beam splitting prism. One beam is coupled to the fiber optic photoelectric antenna (Batop BPCA-100-05-10-1550-C-F) as pump light through a mechanical translation stage, which is used to generate electromagnetic waves. Another beam is coupled to another fiber optic photoelectric antenna (Batop BPCA-180-05-10-1550-C-F) as the detection light, which is used to detect THz waves. A phase change thermoregulation emulsion was injected into the microfluidic chip, placed vertically between two off-axis parabolic mirrors. After passing through the microfluidic chip, the THz wave carries the
information of the sample and is received by the detection antenna, then the signal is amplified by a lock-in amplifier, and finally the data are collected and processed by a computer.

2.2. Development of microfluidic chip
Since COC can reach over 90% transmission of THz waves and is transparent to visible light, the amount of liquid in the chip can be clearly observed, so COC is used as the material for making microfluidic chips. The microfluidic chip in this experiment uses a sandwich structure, as shown in figure 2. First, two COC pieces were cut into rectangles of 4 cm in length, 4 cm in width, and 2 mm in height as the substrate and cover sheet of the chip, and two circular holes of 2 mm in diameter were drilled on the substrate. Then strong double-sided adhesive with a thickness of 50 μm was cut into a concave shape, and the cut double-sided adhesive was used to adhere the substrate and cover sheet together.

2.3. External magnetic field system
The applied magnetic field device used in this experiment is shown in figure 3. Two electromagnets were used to generate the magnetic field. The electromagnets were powered by a WYJ-9B transistorized power supply, and the magnetic field was processed by placing the microfluidic chip between the two electromagnets. THz waves were transmitted to the surface of the microfluidic chip through the central aperture of one electromagnet, transmitted through the microfluidic chip, and then transmitted to the receiver through the central aperture of the other electromagnet. The magnetic field strength at the microfluidic chip was varied by adjusting the output voltage of the transistorized regulated power supply (output voltage range 0–30 V) to vary the operating voltage of the electromagnets.

2.4. External electric field system
The applied electric field device used in this experiment is shown in figure 4, where the material used to fix the microfluidic chip and the metal plate is plexiglass. The device generated a uniform electric field through a high-voltage power supply module (dw-p153-05c51) to two metal plates parallel to each other, and the electric field was processed by placing the microfluidic chip between the two metal plates. By adjusting the sliding resistor, the voltage input to the two metal plates was varied between 0 and 15000 V, thus changing the electric field strength at the location of the microfluidic chip.
2.5. Temperature control system

A temperature control system was designed as shown in figure 5. First, we made a 2 mm thick iron sheet with a 6 mm diameter hole in the middle and a circular alumina ceramic heating plate with an outer diameter of 40 mm and an inner diameter of 10 mm and a rated voltage of 12 V. The temperature sensor and the fabricated microfluidic chip were then fixed on one side of the iron sheet with thermally conductive silicone, and the alumina ceramic heating plate was fixed on the other side of the iron sheet. The heating plate and the temperature sensor were controlled by a temperature controller (ST700 intelligent PID temperature controller: rated voltage: 220 V; operating frequency: 50–60 Hz; accuracy: 0.1 °C). During the experiment, the heating plate was giving continuous heating to the iron sheet, which would then transfer the heat to the microfluidic chip.
allowing the phase change temperature regulation emulsion inside the chip to rise to the specified temperature, and the holes of both the iron sheet and the heating plate were to facilitate the passage of THz waves. During the subsequent cooling process, the sensor displayed the current temperature of the iron sheet at times, thus reflecting the temperature change of the phase change thermoregulation emulsion.

3. Experiment

The PCT emulsion we used in this experiment is made of natural paraffin, which is a class of saturated hydrocarbons without branched carbon chains. It consists of a mixture of n-alkanes having the chemical formula C\textsubscript{n}H\textsubscript{2n+2}, where \( n \approx 20-40 \), and there are no hydrogen bonds between the molecules.

3.1. THz spectral properties of a PCT emulsion at different magnetic field strengths

First, a microfluidic chip containing a 50 \( \mu \text{m} \) thick film of the PCT emulsion was placed in the THz-TDS system. Then we applied a magnetic field perpendicular to the plane of the microfluidic chip, and varied the field strength by changing the voltage on the electromagnet. The adjustable range of the magnetic field is 0–100 mT. Although the adjustment range of magnetic field strength is large, the maximum magnetic field strength in this experiment is only 36 mT. This is because the stronger the magnetic field is, the greater the heating capacity of the electromagnet is, which will increase the temperature of the microfluidic chip and affect the measurement results. The voltages were made to be 9, 12, 15, and 18 V, which means magnetic field strengths of 18, 24, 30, and 36 mT, respectively, and the tests were performed five minutes after applying the magnetic field to ensure that the sample had reached stability in the magnetic field. We disconnected the power supply used to produce the magnetic field for ten minutes after each test in order to eliminate the effect on the next experiment and to restore the sample to its original state as much as possible. After many experiments, we found that this kind of time collocation gets the best experimental data. The experimental results after four such tests are shown in figure 6. Both the time-domain and frequency-domain spectra after Fourier transform show that the THz spectral intensity transmitted through the PCT emulsion increases with the intensity of the applied magnetic field.

To confirm the usability of the above experimental data, three measurements were performed at each magnetic field strength, and the repeatability error was analyzed for 18 mT and 24 mT. As shown in figure 7, the standard deviation of repeated measurements at the same magnetic field strength is smaller than the difference between the two. Therefore, we believe that the measurement deviation has a small effect on the experimental results and the experimental data are valid. Since the emulsion film is very thin, the effect of water on the experimental data is small, and the COC material used to make the chip has a transmission rate of more than 90% in the THz range, and the structure of the material is stable, so it can be argued that the changes in the experimental data are caused by the changes of natural paraffin. All experimental data used in this paper satisfy the above rules.

For further analysis of the experimental data, we calculated the refractive index and absorption coefficient of PCT emulsion according to the method proposed by Dorney and Duvillaret \textit{et al} [16, 17]:

![Figure 6.](image-url) (a) THz time-domain spectra and (b) frequency-domain spectra of paraffin at different magnetic field strengths. The insets show the changes in the peak of the transmitted spectral intensity measured at each magnetic field strength indicated in the legend (a) and (b).
Equation (1) is used to calculate the refractive index of the sample, where $c$ is the speed of light, $\omega$ is the angular frequency, $d$ is the thickness of the sample, and $\psi(\omega)$ is the phase difference between the sample signal and the reference signal. Equation (2) is used to calculate the absorption coefficient of the sample, where $\rho(\omega)$ is the amplitude ratio of the sample signal to the reference signal. The refractive index and absorption coefficient of PCT emulsion at different magnetic field strengths were obtained using the above equations, as shown in figures 8 and 9. It can be seen that the refractive index gradually becomes larger and the absorption coefficient gradually becomes smaller with the increase of the applied magnetic field strength. This is consistent with the trend observed in our experiments that the THz spectral intensity transmitted through the PCT emulsion increased with the intensity of the applied magnetic field.

All the experimental data in this paper are treated as above, and the conclusions obtained are consistent with the observation in the experimental part, so the data under different magnetic field intensities are only used for illustration.

\[
\begin{align*}
n(\omega) &= \frac{c}{\omega d} \psi(\omega) + 1 \\
\alpha(\omega) &= 2 \frac{\ln \left( \frac{4n(\omega)}{\rho(\omega)[n(\omega) + 1]^2} \right)}{d}
\end{align*}
\]
We also investigated the THz transmission intensity, sample refractive index and sample absorption coefficient versus the applied magnetic field strength at a certain frequency. As shown in figure 10, the data at 0.45 THz were selected for linear fitting. The correlation coefficients are 0.98911, 0.95652, and 0.98928, respectively, with good linearity. Therefore, the THz transmission intensity, sample refractive index and sample absorption coefficient can be measured by the THz-TDS technique to deduce the magnetic field intensity to which the sample is exposed.

3.2. THz spectral properties of a PCT emulsion at different electric field strengths
We injected a sample of the PCT emulsion into the microfluidic chip and placed it in the THz-TDS system. Then, 2000, 3000, 4000 and 5000 V cm$^{-1}$ of different electric field strengths were applied in parallel to the microfluidic chip. At each field strength, the sample was applied for five minutes before testing. After each test was completed, we disconnected the power supply used to produce the electric field for ten minutes in order to eliminate the effect of this experiment on the next experiment and to restore the sample to its original state as much as possible. The experimental results we obtained after four such tests are shown in figure 11. The time-domain and frequency-domain spectra both show that the transmitted THz spectral intensity of the PCT emulsion increases with the intensity of the applied electric field, and gradually tends to saturation.

3.3. THz spectral properties of a PCT emulsion at different temperatures
The PCT emulsion was filled into the fabricated microfluidic chip, placed between off-axis parabolic mirrors, and tested its spectral properties using a THz-TDS system. As the temperature is decreased from high to low, n-alkanes with carbon numbers n greater than 20 go through a transition from liquid phase to rotating phase and then to solid phase. Also, different n-alkanes go through different rotating phases at different temperatures. The natural paraffin used in this experiment is a mixture of various n-alkanes, so the experimental results are not
very regular at temperatures greater than 36 °C, where the paraffin may exist in a mixture of liquid, rotational, and solid phases. Therefore, in this paper we focus on the THz transmission properties of paraffin in the range 36 °C–33 °C, when the n-alkanes either are already in the solid phase or else are in the process of transitioning from the rotating phase to the solid phase. We measured the transmission characteristics of the emulsion over the range 36 °C–33 °C, decreasing the temperature by 1 °C between each measurement. The THz time-domain and frequency-domain spectra of the PCT emulsion after four tests are shown in figure 12, which show that the transmitted spectral intensity increases as the temperature is decreased. This experiment and the above two experiments have been repeated three times, and the repeated results of each experiment are the same, which proves the reliability of each experiment.

4. Results and discussion

In a magnetic field, the THz transmission of a PCT emulsion increases as the strength of the applied magnetic field increases. Because the THz transmission intensity of pure deionized water decreases with the increase of magnetic field intensity [18], the changes of time-domain and frequency-domain spectra in figure 6 reflect the changes of paraffin molecules in PCT emulsion. As illustrated in figure 13, the paraffin molecule is a diamagnetic substance, and the intrinsic magnetic moment of each molecule is zero in the absence of a magnetic field. However, in a strong external magnetic field, even though the paraffin molecule has a low magnetization rate, the internal energy is partially changed, and individual electrons rotating around the nuclei in the molecule are
subjected to the Lorentz force. This causes some molecules to produce a magnetic moment oriented in the opposite direction to the external magnetic field, so this component of the paraffin molecules can be considered to be magnetized [19]. These magnetized paraffin molecules produce an induced magnetic moment that changes the original arrangement of the paraffin molecules in the magnetic field, orienting them and increasing their intermolecular spacings [20], which favors the passage of THz waves. Moreover, paraffin molecules tend to align with their poles antiparallel to the magnetic field direction. These weak dipoles thus create a repulsive force between the molecules, and this repulsive force changes the morphological characteristics of the process of crystalline agglomeration [19]. When the strength of the applied magnetic field is gradually increased, more paraffin molecules become magnetized, which results in a more oriented arrangement and larger intermolecular spacings. The THz transmission therefore increases with the magnetic field strength.

In an electric field, the intensity of THz radiation transmitted though the PCT emulsion increases—and the intensity of THz absorption by the emulsion consequently decreases—as the strength of the applied electric field increases. Because the transmission intensity of pure deionized water and PCT emulsion under the same electric field has the same increasing trend, we have made many contrast experiments. It is found that the THz transmission intensity of pure deionized water is lower than that of PCT emulsion at a fixed frequency and electric field intensity. Therefore, we think that with the increase of electric field intensity, the THz transmission intensity of paraffin molecules will gradually increase. This occurs because the orientations of the paraffin molecules in the emulsion are disordered and in equilibrium when no electric field is applied. When there is an external electric field, however, the atoms in the paraffin molecules become unbalanced by the force exerted by the electric field. Consequently, the molecules move, changing the distances between the atoms—and thus the electrostatic repulsion—until they finally reach a new equilibrium, as illustrated in figure 14. When the strength of the applied electric field is increased gradually, the force it exerts also increases gradually, the total energy of paraffin molecules decreases, the strengths of the covalent bonds decrease. In consequence, the bond lengths increase, so the average distance between the atoms increases. The molecular mean free path increases, the molecular motion becomes more free [21], and the THz transmission becomes higher.

As figure 12 shows, the intensity of THz radiation transmitted through the PCT emulsion differs at different temperatures, and it increases as the temperature decreases. Since the THz transmission intensity of pure deionized water also increases with the decrease of temperature, in order to explain the influence of PCT emulsion on the THz transmission intensity with the decrease of temperature, we have carried out many contrast experiments on pure deionized water and PCT emulsion, and found that the THz transmission intensity of pure deionized water is less than that of PCT emulsion at the same temperature. Therefore, we believe that paraffin molecules have an influence on the THz transmission intensity. During cooling, the vibrations of the n-alkane molecules weaken, leading to a decrease in the free energy, and the crystal structure of
the n-alkane molecules already in the solid phase develops in an ordered and stable direction, while the alkanes in the rotating phase continue to undergo crystallization. Although the crystal structure of the rotating phase lacks long-range rotational order, it causes crystallization gradually to align neatly until it reaches a state of relative equilibrium. In this process from disorder to arrangement and order, the scattering of THz waves is reduced, so there is an increase in the intensity of THz transmission [22, 23].

5. Conclusions

In conclusion, we have combined THz technology and microfluidic technology to study the THz transmission characteristics of the PCT emulsion paraffin subject to changes in the external environment. We found that as the strength of an external magnetic field is increased, more molecules are magnetized and they become arranged more directionally. Further, as the strength of an external electric field is increased, the total energy of paraffin molecules decreases, the average interatomic spacing increases. In addition, the molecular vibrations decrease and the arrangement becomes more orderly as the temperature is decreased. All three environmental changes modify the internal structure of the PCT emulsion, resulting in an increase in the transmitted intensity of THz radiation. Therefore, THz techniques can be used to analyze the properties of PCT emulsions by measuring the THz transmission intensity, refractive index, or sample absorption coefficient to deduce the environment to which the sample is exposed. This study also provides data support for further in-depth investigations of these materials. It is important for the fine research and improvement of the performance under different intensity magnetic field, different intensity electric field and different temperature in aerospace and coating fields.

Acknowledgments

This work is supported by the National Key R&D Program of China (Grant No.2021YFB3200100) and National Natural Science Foundation of China (NSFC) (61575131). The authors would like to thank ‘Enago’ for providing English touch up.

Data availability statement

The data that support the findings of this article are available upon reasonable request from the authors.

ORCID iDs

Bo Su  https://orcid.org/0000-0003-1851-2621

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