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Terahertz absorption characteristics of guar gum determined via microfluidic technology

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Abstract

The vibrational energy levels of many biomolecules correspond to the terahertz band; thus, terahertz technology can be used to identify these substances. Moreover, as the biological activity of most biomolecules can be observed only in aqueous solution, the characteristics of such biomolecules must be studied in aqueous solution. In this study, a simple microfluidic chip, a temperature control device and a strong electric field device were designed to study the terahertz absorption characteristics of guar gum for different temperatures, concentrations and electric field exposure durations, thus enabling the use of terahertz technology to analyse the characteristics of guar gum.

Keywords: Terahertz, Guar gum, Microfluidic chip, Temperature, Concentration, Electric field

Introduction

Electromagnetic waves in the terahertz (THz) band have a frequency range of 0.1–10 THz and a wavelength range of 0.03–3 mm. Studies have shown that the characteristic vibration modes of many biological macromolecules correspond to the THz band [1]. Consequently, THz waves can be used to detect biological molecules [2–6]. Water plays an important role in the function of biomolecules, and thus, most experiments are performed in the solution state. Interactions among hydrated molecules in solution involve multiple biological phenomena [7], which are often denoted as hydration. Based on this principle, Yang et al. measured the spectrum of L-asparagine and L-asparagine monohydrate via THz time domain spectroscopy (THz-TDS) technology. In addition, he also observed dynamic changes in L-asparagine thermal dehydration in real time and found that THz waves are highly sensitive to changes in the crystal structure, the condition of the solution in which the crystal is contained and weak interactions between molecules [8]. There are many applications of terahertz such as investigation of non-metal samples such as plastic, semiconductors, and in medicine. M. Yamagiwa et al. applied angular spectrum method (ASM) to the acquired THz digital hologram, and performed real-time amplitude/phase imaging and digital focusing of visibly opaque objects. The demonstrated real-time and precise 3D imaging capability will be a powerful tool for the non-destructive inspection of optically opaque soft materials [9]. In order to improve the steepness of the thick edge of the film in terahertz off-axis digital holography, D. G. Abdelsalam has carried out experiments with terahertz quantum cascade laser (THz-QCL) and terahertz cameras. The results show that the direct Fourier and Hermite polynomial reconstruction method is very promising [10]. THz rays have higher penetration depth compared to visible rays, so D. G. A. Ibrahim et al. have measured the individual thickness of adhesive layers on multilayered adhesive structure comprised of thick and thin polymer films with the terahertz pulsed imaging (TPI) system [11]. Microfluidics, also known as lab-on-a-chip or microfluidic chip technology, enable

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researchers to precisely control and manipulate micro-scale fluids with submicron structures. Thus, microfluidics have practical value for distinguishing similar substances and detecting trace samples. Fisher et al. measured the optical constants of four nucleotides in the THz band using THz technology and successfully identified these nucleotides [12]. Serita et al. developed a THz microfluidic chip based on a nonlinear optical crystal, comprising an array of several split ring resonators, for the detection of ultra-trace biological samples [13]. Baragwanath et al. used THz-TDS to examine a microfluidic chip and found that the time domain spectrum, refractive index and other parameters differed greatly for different concentrations and substances [14]. Thus, THz spectroscopy technology is a powerful tool for distinguishing substances and studying their structures.

Guar gum is an emulsifier, curing agent and stabiliser. Guar gum can act as a thickening liquid, can increase viscosity and reduce ice crystal formation, and is used in most processed foods. In guar gum solution, guar gum molecules will interact with water molecules to form hydrogen bonds. It is well known that hydrogen bonds in solution will have a strong absorption effect on terahertz, so it is very meaningful to study guar gum solution. On the other hand, the absorption of guar gum solution to THz is different under different conditions, such as different concentration, different temperature, external electric field or external magnetic field. In this study, THz technology and microfluidic approaches were combined to study the THz absorption characteristics of guar gum for different concentrations, temperatures and electric field treatment durations. The method presented herein provides a feasible approach for rapidly identifying colloids.

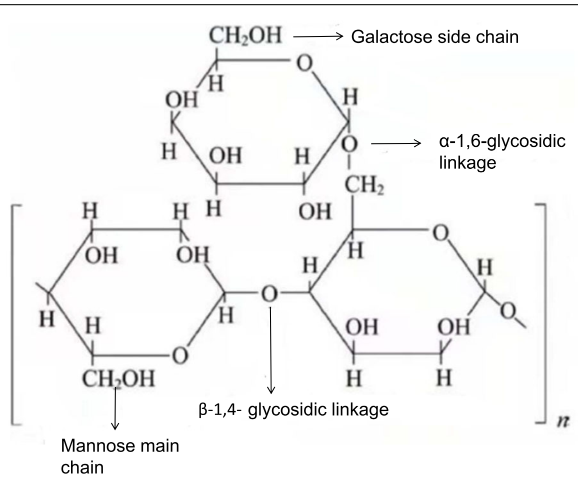

Fig. 1 Molecular structure of guar gum

Properties of guar gum

The molecular structure of guar gum is shown in Fig. 1 [15]. Guar gum is extracted from the endosperm of the leguminous plant guar bean as a free-flowing powder. The powder is generally white to light yellowish brown in colour and is nearly odourless. Guar gum is composed of approximately 75%–85% polysaccharide, 5%–6% protein, 2%–3% fibre and 1% ash.

Guar gum is a non-ionic neutral polysaccharide, with D-mannose linked by a β-1,4 bond as the main chain and D-galactose linked at the α-1,6 position as the branch chain. In general, the molar ratio of galactose to mannose is 1:2. The spatial structure of the plant is a curled spherical structure, with mannose on the inside and galactose on the outside. The ratio of mannose to galactose varies slightly for different plant varieties.

Guar gum is a type of water-soluble natural polymer, which can be quickly hydrated in cold or hot water to obtain a translucent viscous liquid. The viscosity of a guar-gum-containing aqueous solution reflects the molecular weight of guar gum, while the swelling rate reflects the degree of difficulty that is encountered in swelling in guar gum applications. Higher temperatures can accelerate the swelling of guar gum, weaken the hydrogen bonding between molecular chains, increase the elongation of molecular chains and reduce the viscosity.

Notably, guar gum can form a high-viscosity aqueous solution at low concentrations due to entanglement between polymer chains and intramolecular and intermolecular hydrogen bonding. The viscosity of 1% guar gum in aqueous solution is approximately 4000–6000 MPa·s. In addition, guar gum aqueous solutions exhibit typical winding polymer characteristics, corresponding to the pseudoplastic fluid characteristics of a non-Newtonian fluid, with no yield stress. Because guar gum is widely utilized and exhibits a high viscosity and rheological properties in natural rubber, the THz absorption of guar gum was studied in this work, with guar gum acting as a representative colloid for further experimental research.

Experimental system

Experimental light path

An in-house-built THz-TDS system was employed in this work. The light source is a self-mode-locked fibre femtosecond laser purchased from Peking University, with a central wavelength of 1550 nm, pulse width of 75 fs, pulse repetition rate of 100 MHz and power of 130 mW. After passing through a polarising beam splitter prism, the output laser is divided into two beams. One beam is used as the pump light, which is coupled into a fibre-optic photoconductive antenna (BPCA-100-05-10-1550-c-f, BATOP Company) through a mechanical translation stage to generate a THz wave. The other beam is used as a detection beam, which is coupled into another fibre-optic photoconductive antenna (BPCA-180-05-10-1550-c-f of BATOP Company) to detect THz waves. The fabricated microfluidic chip is placed between two off-axis parabolic mirrors. The THz wave emitted by the THz antenna passes through the microfluidic chip filled with guar gum, is received by the detection antenna and is then input into the lock-in amplifier for amplification. Next, the data are collected and processed by a computer. A schematic of the experimental optical path is shown in Fig. 2.

Fabrication of the microfluidic chip

THz microfluidic chip materials should exhibit high transmission and allow for ease in processing. Currently, microfluidic chips are generally made of quartz glass, polydimethylsiloxane (PDMS) or polyethylene (PE). However, due to the low transmission of quartz glass and PDMS for THz waves and the opacity of PE to visible light, these materials do not allow one to observe changes in liquid samples in a channel in real time. The cycloolefin copolymer zeonor 1420R has a very high THz transmission and no absorption peak. The THz transmission of 2 mm thick zeonor 1420R is shown in Fig. 3. It can be seen that the terahertz transmission of the material is more than 90%, which is an ideal material for preparing terahertz microfluidic chips.

The microfluidic chip in this study is a sandwich structure, with a 50-μm-thick strongly adhering double-sided adhesive as an interlayer and a concave microfluidic channel carved in the middle. The substrate and cover of the microfluidic chip are made of zeonor 1420R with a thickness of 2 mm. The length and width of the whole chip are 3 cm and 2 cm respectively. The thickness of double-sided adhesive determines the depth of microfluidic channel and the thickness of guar gum solution. The diameter of liquid inlet and outlet is 2 mm. A diagram of the process for preparing the microfluidic chip is shown in Fig. 4.

In this study, we first measured the THz transmission characteristics of the microfluidic chip during continuous heating without liquid injection in order to assess the THz transmission characteristics of the chip under varying temperature. The results show that the transmission characteristics of the chip are not affected by temperature and that there is no leakage phenomenon. Therefore, the chip is suitable for the detection of liquid samples at different temperatures.

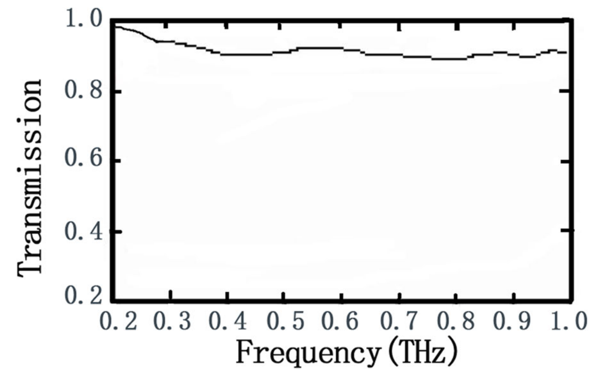

Fig. 3 Transmission of Zeonor 1420R

Temperature control system

To control the temperature of the guar gum, a high-precision temperature control system was designed. First, the fabricated microfluidic chip was bonded to a 2-mm-thick iron sheet by using a thermal conductive silica gel. A 6-mm-diameter hole was created on the iron sheet for THz waves to pass through. Second, a temperature sensor was secured to the same side of the iron sheet using thermal conductive silica gel. A circular alumina ceramic heating plate with holes (outer diameter: 40 mm; inner diameter: 10 mm) was glued to the other side of the iron sheet using thermal conductive silica gel in order to heat the microfluidic chip. The heating plate and temperature sensor were controlled by a temperature controller (ST700 intelligent PID temperature controller; temperature adjustment range:

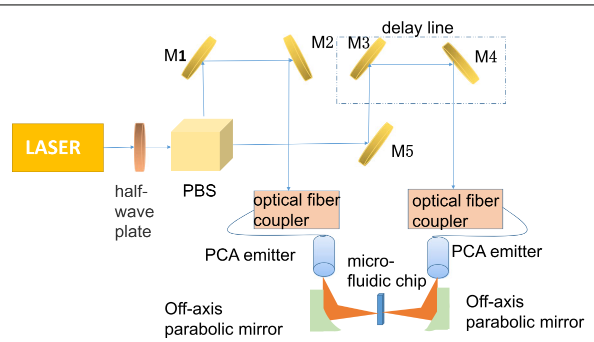

Fig. 2 Diagram of the experimental light path

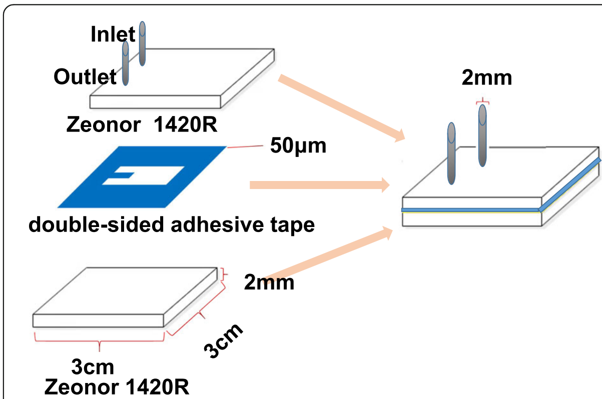

Fig. 4 Fabrication of the microfluidic chip

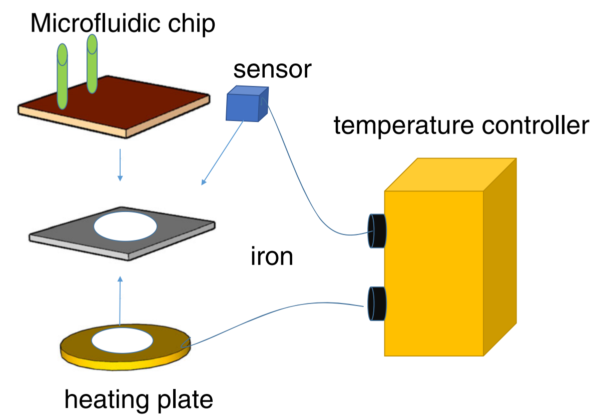

Fig. 5 Schematic diagram of the temperature control system

0–400 °C; rated voltage: 220 V; working frequency: 50–60 Hz). The temperature controller, which is shown in Fig. 5, can adjust the temperature with an accuracy of 0.1 °C.

External electric field device

In colloidal dispersion system, colloidal particles can often adsorb ions from the medium, so that the dispersed colloidal particles are charged. Different colloidal particles have different surface composition. Some of them can absorb positive charge, others can absorb negative charge. Therefore, some colloidal particles have positive charge and some negative charge, and they will move in different directions under the action of electric field. Therefore, it is considered that the change of colloidal internal structure may lead to the change of terahertz absorption characteristics.

A schematic diagram of the external electric field device utilised in this experiment is shown in Fig. 6. A high-voltage power supply (dw-p153-05c51) was used to provide power. A uniform electric field was generated by two parallel metal plates, with a magnitude of approximately 2500 V/cm. The microfluidic chip was placed between the two metal plates for electric field treatment; subsequently, the two metal plates were removed, and the transmission of the microfluidic chip was measured by the THz-TDS system.

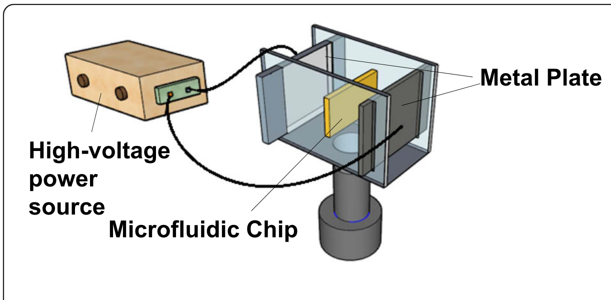

Fig. 6 Schematic diagram of the external electric field device

Experiments and results

Temperature characteristics

In this experiment, guar gum was prepared by adding 0.01 g guar gum powder to 500 mL deionised water, then, the mixture was stirred evenly and heated in a water bath for 2 min, resulting in 0.002% guar gum solution. When the colloid was irradiated with a laser pen, an obvious Tyndall effect was observed, indicating that the solution was a sol. The microfluidic chip was filled with the guar gum solution and placed between off-axis parabolic mirrors to conduct a transmission test using the THz-TDS system. The transmission at room temperature (25 °C) was measured first, followed by the transmission at 30 °C to 70 °C, measured in increments of 10 °C. When the temperature reaches 60 °C, the colloid inside the chip produces bubbles due to the temperature rise, and the measured signal fluctuates. To reduce the influence of bubbles on the measured signal, guar gum solution was continuously injected into the microfluidic chip when bubbles occurred during the heating process, and the solution was injected until the bubbles in the chip were eliminated. In this process, the colloid in the chip heats rapidly. When the guar gum solution reaches the target temperature, there are fewer bubbles in the water, which will not affect the analysis of the measured data. In order to ensure the stability of the measured signal, each temperature gradient is repeated for at least four times. In addition, all the experiments in the follow-up of this paper also carry out the same operation. The THz time domain spectrum of guar gum is shown in Fig. 7. It can be found from the figure that the higher the temperature is, the lower the peak value is, and the peak value moves to the right. This is because the higher the temperature, the stronger the interaction

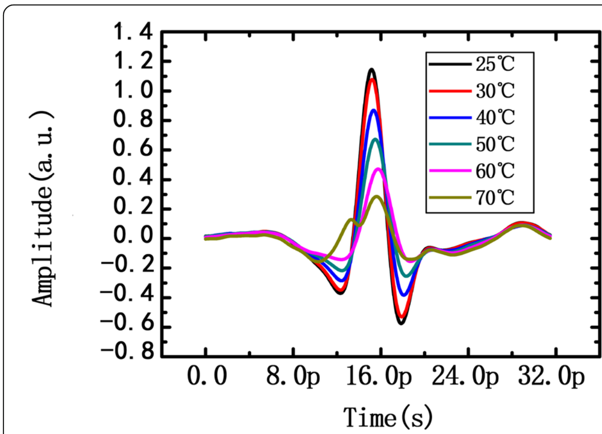

Fig. 7 THz time domain spectra of guar gum at different temperatures

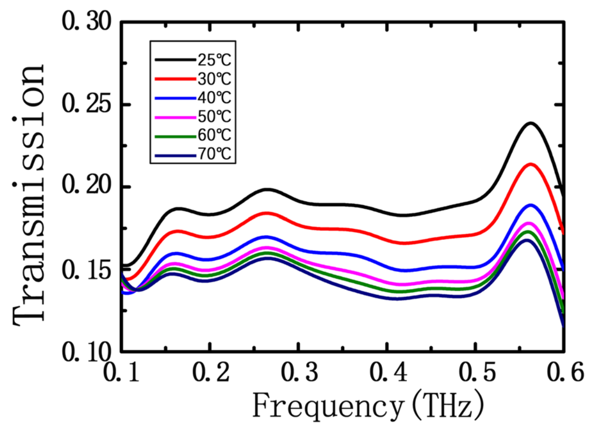

Fig. 8 THz frequency domain spectra of guar gum at different temperatures

between guar gum molecules and water molecules, and the stronger the absorption of terahertz wave. The higher the temperature is, the higher the viscosity of guar gum is. Therefore, when THz wave passes through, the optical path increases, the transmission time becomes longer, and the peak value shifts to the right.

The frequency spectrum obtained by Fourier transform is shown in Fig. 8. The results shown in the figure demonstrate that the THz transmission intensity of guar gum decreases with increasing temperature, indicating that the THz absorption of guar gum increases with increasing temperature in the range of 25 °C–70 °C.

Concentration characteristics

Three concentrations of guar gum solution (0.1%, 0.2% and 0.3%) were prepared with deionised water. The THz frequency spectra for these solutions are shown in Fig. 9. These results indicate that a higher concentration of guar gum solution corresponds to a lower THz transmission intensity, resulting in a stronger THz absorption.

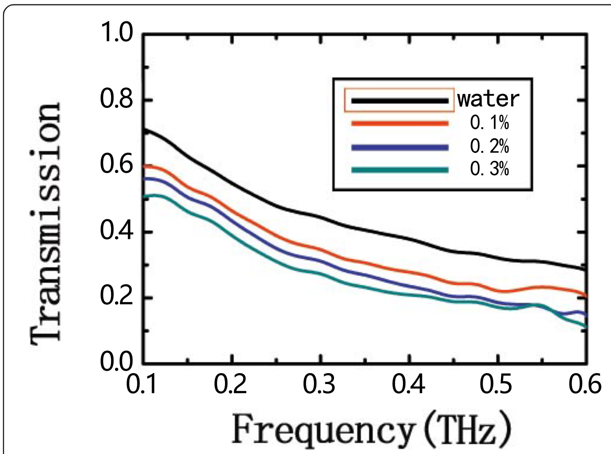

Fig. 9 Spectra for different concentrations

Characteristics under an applied electric field

The 0.002% guar gum solution was injected into the microfluidic chip, and the THz spectrum was obtained via the THz-TDS system without an electric field. Then, an electric field of 2500 V/cm was applied parallel to the microfluidic chip, and the guar gum solution was placed in the electric field for 5 min. The power supply was then disconnected, the electrode plate was removed, and the THz spectrum was obtained with the THz-TDS system. Next, the microfluidic chip was shaken to return the sample to the original state as much as possible, the sample was placed in the electric field for 10 min, and the THz spectrum was obtained again. Based on the above operation steps, we measured THz transmission characteristics at 15 min, 20 min and 25 min. The THz spectra obtained in these five measurements are shown in Fig. 10. The results show that the THz transmission increases with an increasing duration of electric field exposure, implying that the THz absorption intensity of the guar gum solution decreases.

Discussion

The THz spectra of guar gum solution at varying temperatures show that the THz transmission decreases with increasing temperature. This result occurs because the outer layer of hydroxyl, alkoxy and oxygen atoms in glycoside bonds in guar gum molecules containing sp^3 hybrid orbitals. The unshared lone electron pairs in the orbitals can combine with partially positively charged hydrogen in the water molecule to form hydrogen bonds through electrostatic attraction [16]. Due to the stretching of guar gum molecules, a variety of groups are fully exposed. The polar groups and polar water molecules interact with each other by hydrogen bonding or dipole forces to form an inner-layer water film. The inner-layer water reacts with the outer-layer water to form an

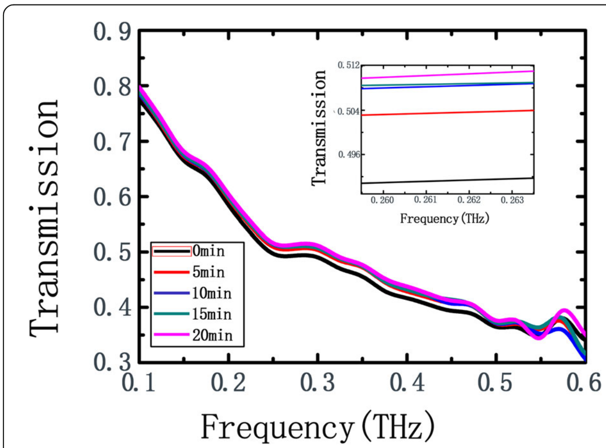

Fig. 10 Frequency domain spectra of the THz transmission intensity of guar gum for different durations of electric field exposure

association. The sol molecules with a large volume act as the skeleton, a large amount of water is bound, and free movement of the medium is hindered, resulting in resistance between laminar flows. This resistance is exhibited as apparent viscosity, which causes the solution to display a quantitative viscosity [17]. When the temperature rises, the rate of molecular motion gradually increases, and the aggregates change from large to small. In this process, the oxidative degradation of guar gum molecules leads to hydrogen bond breaking [17], and the THz transmission intensity should be enhanced. However, guar gum solution is a polar liquid in which dipole interactions occur. The absorption coefficient of polar liquids is larger in the THz range approximately 10–100 times that of non-polar liquids [18]. Therefore, the vibration between dipole molecules is the main reason that the THz absorption of guar gum increases with increasing temperature.

The THz transmission spectra of guar gum solution for varying concentrations (Fig. 9) show that the transmission intensity of THz waves decreases with increasing concentration, consequently, the THz absorption of guar gum solution increases. This is because with the increase of guar gum concentration, the number of polymer molecular chains relative to the solvent increases, and the molecular chains are intertwined due to insufficient extension, which increases the force of molecular chains and thus shows higher viscosity. When the viscosity increases, the dispersed phase in the system is not easy to aggregate and agglomerate, so the dispersion system can be stabilized [19]. Due to the strong intermolecular hydrogen bond network and the influence of intramolecular vibration mode, the THz absorption of guar gum solution increases with the increase of concentration.

The THz transmission spectra of guar gum solution exposed to an electric field for varying durations (Fig. 10). The results show that longer time exposure to electric field will lead to higher THz transmission intensity of guar gum solution. In the absence of an external electric field, the orientation of the molecules in the solution is disordered, and the optical anisotropy effect of each molecule is counteracted. Under the action of the electric field, the molecules are aligned along the direction of the electric field, which further increases the rigidity of the molecular chain [20]. With the increase of applied electric field time, the transmission of THz is also increasing, which may be due to the effect of electric field hindering the cross-linking and aggregation of colloids, and the side chain of guar gum hindering the aggregation of its own macromolecules, reducing the intermolecular hydrogen bonding. Therefore, the absorption of THz decreases with the increase of exposed time.

Conclusion

In this paper, a new method of making THz microfluidic chip is designed. Zeonor1420R is used as the substrate and cover, and double-sided adhesive is used as the channel interlayer of microfluidic channel, which greatly shortens the fabrication time and resolves the issue of chip leakage. Meanwhile, in order to control the temperature change, a temperature control system with high precision is designed, then the transmission characteristics of guar gum solution at different temperatures are measured by the system. It can be seen from the time domain spectrum that the shift of THz pulse appears when the temperature rises to 30 °C, it is possible that the shift is caused by the internal space effect and the weak intermolecular interaction [21]. It can be seen from frequency spectrum that with the increase of temperature, the transmission strength of THz is lower and lower, mainly due to the interaction between dipole molecules in the polar liquid guar gum solution. Then, the different concentrations of guar gum solution were studied, compared with deionized water, the THz transmission intensity of guar gum solution decreased with the increase of concentration, which was mainly due to the strong hydrogen bond network and intramolecular vibration mode between molecules, resulting in the increase of THz absorption. Finally, and a high-voltage power supply was used to provide a strong electric field, the THz transmission characteristics of guar gum at different times were detected, compared with no electric field, the THz transmission increases with the increase of electric field time. Under the action of electric field, molecules are arranged along the direction of electric field. With the increase of electrifying time, the side chain of guar gum hinders the aggregation of its macromolecules, resulting in the weakening of THz absorption.

THz wave has strong penetrating power and is very sensitive to the change of molecular dipole moment. This study provides some guidance for the application of guar gum, expands the application field of THz technology, and lays a foundation for the study of colloidal properties by THz technology.

Abbreviations

PBS: Polarisation beam splitter; PCA: Photoconductive antenna; PID: Proportion integration differentiation; ASM: Angular spectrum method; THz: Terahertz; THz-TDS: THz time domain spectroscopy; THz-QCL: Terahertz quantum cascade laser; TPI: Terahertz pulsed imaging

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Authors' contributions

Feng-Xuan Zhang is the main author of this manuscript. She has carried out experiments and simulations with Guo-Yang Wang and Hai-Yun Huang for many times. Meng-Han Chen and Si-Jia Zhang have consulted relevant literature. Bo Su is the corresponding author of this manuscript. Bo Su and Cun-

Lin Zhang have provided academic guidance for this manuscript. The authors read and approved the final manuscript.

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Availability of data and materials
Information about data was detailed in the article.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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