

Invited Paper

Quantitative Water Content Measurements in Food Wafers Using Terahertz Radiation

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Abstract: A quantitative, non-invasive method of measuring the moisture content (< 25 wt %) of solid food wafers is presented. The method utilizes terahertz (THz) radiation and exploits its high sensitivity to small amounts of water. A linear relationship between transmitted THz signal intensity and moisture content is observed in the range 0.2-0.6 THz. Higher frequencies are affected by scattering due to the size of the pores in the food wafer. A robust quantitative measurement protocol, which requires simple calibration, is implemented based on a direct interpretation of the THz time domain signal.

Keywords: Spectroscopy, THz, Moisture determination, Food wafers

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1. Introduction

Measuring moisture content accurately is of utmost importance to the food processing industry. Moisture is related to many properties that affect the quality and the shelf-life of the final food product, including bacterial growth and texture. This problem becomes even more acute when multi-domain foods are considered as the difference in water activity between the different domains can also result in moisture migration until an equilibrium is reached within the system [1]. Unwanted moisture migration can affect microbial stability, physical and sensory properties and the rate of chemical changes of a food system that essentially reduce its shelf-life.

In the confectionery industry starch wafers are very common and are found in a variety of products such as chocolate bars, biscuits and ice-creams. Wafers are generally low moisture food products and have a porous structure. Their texture and mechanical properties are critical characteristics [2, 3]. Once the non-aqueous ingredients have been chosen, the mechanical properties of the wafers depend predominately on the moisture content. This is due to the plasticization effect of water in the starch matrix [3]. If for instance the baked product is dry, then it is brittle and breaks when it is driven onto cutting blades before the final product is produced. Consequently a lot of the material needs reprocessing or is wasted. If the moisture content is too high then the texture is affected and bacterial growth results in significant decrease of the product shelf-life. Since this is a multi-million pound industry in the UK alone [2], quantitatively monitoring the moisture content of wafers at several stages in the production line is very important.

In order to measure such food characteristics in a non-destructive way, several spectroscopic techniques have been explored [4]. The moisture content of systems similar to the one under study here have been investigated with Nuclear Magnetic Resonance (NMR) [5, 6], Magnetic Resonance Imaging (MRI) [7, 8], Near Infrared (NIR) spectroscopy [9] and Microwave radiation [10].

On-line monitoring of the moisture content in wafer production lines is usually performed by means of NIR spectroscopy [2], which is both a non-invasive and fast measurement. Typical values of the absorption coefficient of water in the NIR region are of the order of $2.6 \times 10^{-2} \text{ cm}^{-1}$ [11]. NIR spectra of foods comprise of broad bands that arise from overlapping absorptions, mainly from overtones and combinations of vibrational modes involving C-H, O-H and N-H chemical bonds. The concentrations of food constituents such as water, protein, fat and carbohydrates can, in principle, be determined by using such absorption spectroscopy. The main drawback of this method arises from the complexity of the data. Multivariate mathematics needs to be applied in parallel with calibration and empirical model fitting [9, 12]. Since several ingredients of the food system can absorb NIR radiation, a change in the ingredients requires a complete recalibration of the measurement. In a versatile food production line where different ingredients are used for different products, the task of recalibration becomes very challenging, while slight alterations in the raw materials can also affect the measurement.

Microwave radiation is an alternative to NIR spectroscopy for such online measurement applications [10, 13, 14]. Typical absorption coefficients for water in the microwave region of the electromagnetic spectrum are of the order of 1 cm^{-1} [15]. Since water absorbs quite strongly in this range of the electromagnetic spectrum, techniques employing microwave radiation can give accurate measurements with good signal-to-noise. The high output power of the generators used in this case however, which typically is of the order of tens of milliwatts [16], can sometimes result in excessive heating and degradation of the sample.

The part of the electromagnetic spectrum between microwaves and the infrared, referred to as terahertz (THz, 10^{12}Hz) radiation, has been used recently for measuring water content differences in several low water content systems. Terahertz radiation penetrates most dry and non-polar substances [17]. In contrast polar liquids are almost opaque to terahertz. Water as a polar molecule absorbs terahertz radiation very heavily [18] and the spectral properties of water at terahertz frequencies under various conditions are well known [19]. Mittleman, Jacobsen and co-workers demonstrated the potential of terahertz radiation for measuring the moisture content of common houseplant leaves [17] and plants [20]. Terahertz radiation has also been used to follow moisture content changes in paper [21], as well as production faults in plastic packages based on the absorption of terahertz radiation by water filled pores [22]. It has also been used for printing evaluation [23], where the thickness of the ink as well as the moisture level can be simultaneously measured. In food system applications, terahertz time-domain spectroscopy (THz-TDS) has been used to measure the moisture content of crushed wheat grains [24].

Measurement techniques using terahertz radiation have certain advantages that can be exploited for measuring the moisture content in food samples. As mentioned earlier, water strongly absorbs terahertz radiation, with typical absorbance values of the order of 250 cm^{-1} [18]. Other constituents of food wafers such as proteins show typical absorption coefficients of the order of 2 cm^{-1} [25] and fats and lipids present typical values of 15 cm^{-1} [26, 27]. Starch, which is the main constituent of wafer, has an absorption coefficient of 6 cm^{-1} in this part of the

electromagnetic radiation spectrum [25]. The above absorption coefficients were all measured between 0.1 – 0.5 THz. Hence, measurements employing terahertz radiation are, in the absence of significant scattering, dominated by absorption due to moisture content and are relatively immune to small fluctuations in the concentration of other constituents of the food sample. Another advantage of this method is the minimal power used for THz-TDS measurements (typically below 10 μW), which does not result in any significant heating of the sample. THz-TDS can thus be considered both as a non-invasive and non-intrusive method.

In this paper we present a method for determining the moisture content of intact food wafer samples by using terahertz transmission measurements. The method is simple, robust and requires minimal calibration as terahertz absorption is dominated by water content. We also briefly discuss the possible extrapolation of this method onto mobile, cheaper hardware and its potential for rapid on-line measurement.

2. Materials and Method

2.1 Wafer Samples

The samples used in this study are rectangular pieces of intact wafer provided by Nestlé, a photograph and cross-sectional schematic of which are shown in Fig. 1. Wafers are essentially starch foams produced by depositing wheat flour batter and heating it between two plates [2]. The two metal plates have an engraved pattern to ease the removal of the wafer sheet once it is baked. This produces the reeding pattern on the surface of the wafer, as seen in Fig. 1. In addition, these structures also contribute to the overall texture of the finished product. The flour is predominately composed of starch with a small amount of gluten and water. Yeast and sodium bicarbonate are used as aerating agents. Different types of starch can be used for different products depending on the final desired properties. The wafers are highly porous structures (typically 80 – 90 % porosity). There is a large distribution of pore sizes across the cross-section of the wafer, with the larger pores being of the order of hundreds of micrometers, and which are located mostly in the centre of the sample, while the smallest pores, of the order of a few micrometers, are located predominately near the edges. High resolution Scanning Electron Micrograph (SEM) and Confocal Laser Scanning Microscopy (CLSM) images of such wafers have been published elsewhere [2] and illustrate this typical pore size distribution in the cross-section of the samples.

The wafer samples were initially dried in a dry nitrogen environment over a period of several weeks to ensure evaporation of the free water; at that point only water that is bound in the starch matrix was present in the samples. The samples were then exposed to moist air for variable time periods followed by a THz-TDS transmission measurement. After the terahertz measurements, the samples were placed in a Sartorius MA-45 moisture analyser (Sartorius AG, Göttingen, Germany) where the exact moisture content was measured and the initial bound moisture content was determined. This moisture analyser is equipped with a motorised ceramic heater generating infrared (IR) radiation and uses gravimetric analysis to determine moisture loss. The resolution of the balance is 0.1 mg. The accuracy of the moisture analysis with regards to small samples masses (< 200 mg) has been reported to be $\pm 0.2\%$ [28]. Two different sets of samples were used in this study covering the lower and higher end of porosity found in a typical wafer production line. Type I is a denser type of wafer with an overall porosity of 77 vol %, while

Type II is the lighter wafer sample with porosity of 88 vol %. Type I and II wafers are produced from a different type of raw materials and the density of the solid matrix in both cases is different. The solid matrix in Type I is roughly twice as dense as in the case of Type II. In addition in-situ drying of Type I wafer was periodically monitored with a THz-TDS transmission measurement.

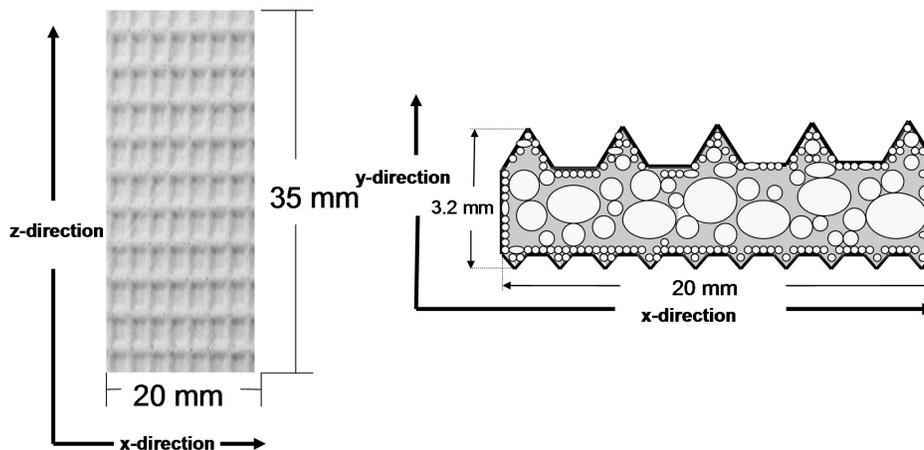


Fig. 1 (a) A photograph of a wafer sheet with dimensions shown. (b) Schematic representation of a cross-section of a wafer sample through which the THz measurements are taken. The typical porosity distribution, the reeding pattern and the dimensions are shown.

2.2 Flow-cell

A flow-cell (Fig. 2) was designed for in-situ terahertz transmission measurements. The cell is sealed from the dry nitrogen environment of the sample chamber where the terahertz optics are located, and hence prevents the samples from drying. A gas flow (nitrogen or air) of controlled flow-rate, temperature and relative humidity is connected to the inlet of the cell at the top. The conditioned gas then flows vertically through the cell and leaves the cell through the outlet at the bottom. For the drying experiments performed in this work, dry nitrogen was blown at ambient temperature (18 °C) through the cell. For all measurements the terahertz beam is transmitted through a set of polytetrafluoroethylene (PTFE) windows sealing the flow cell, which are virtually transparent to terahertz radiation. Different sample holders can be used depending on the size and shape of the sample under investigation. The sample holder used in this study is shown in Fig. 2. It has a square aperture to fit the wafer samples. The cell is designed such that it is possible to easily move the sample out of the beam path in order to acquire a reference measurement through the empty flow-cell.

2.3 Terahertz spectrometer

The setup used for the acquisition of the terahertz time-domain signal has been described previously [29, 30]. Sub-picosecond coherent pulses of broadband terahertz radiation (0.1 - 4 THz) were generated by photo-excitation of a DC biased semi-insulating GaAs substrate by 12 fs

pulses of a NIR laser (Femtolasers, Femtsource cM1, Vienna, Austria, centre wavelength 800 nm). Using two parabolic off-axis mirrors the terahertz pulses were focused close to the samples and the transmitted pulses were collected using an identical set of parabolic mirrors. A coherent detection scheme was used employing electro-optical sampling. This approach allows for the measurement of the terahertz electric field directly rather than just its amplitude. To increase the speed of data acquisition as well as to improve spectral resolution and signal-to-noise ratio, a 50 ps rapid optical delay was used. Between 200 and 300 scans were co-added at a scanning frequency of 0.5 Hz for each spectrum. Similar systems using a rotary optical delay line report scanning frequencies up to 400 Hz with comparable signal-to-noise ratio [31], thus acquisition times can be reduced to a second.

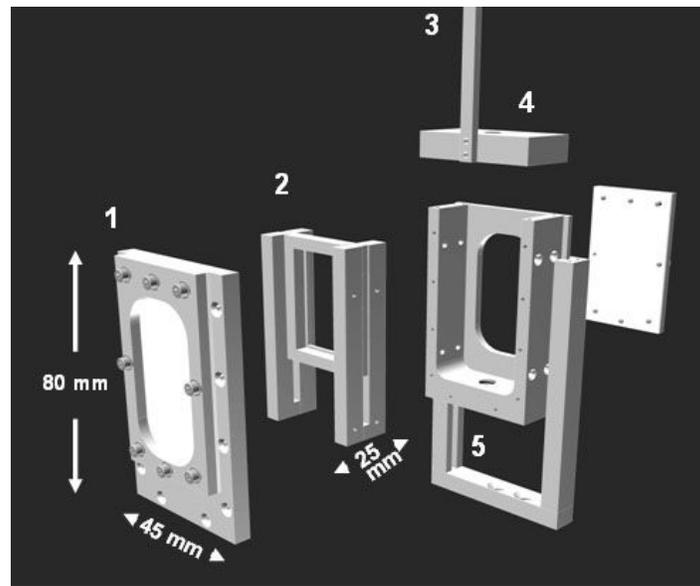


Fig. 2 Design of the flow-cell. (1) PTFE window, (2) sample holder that can be modified depending on the sample size, (3) holder that allows the sample to be removed from the optical path in order to acquire a reference measurement, (4) flow inlet, (5) flow outlet.

The acquired time-domain waveform was apodized using a Hamming function. To account for laser fluctuations during the measurements, the time-domain transmission signal of the samples is normalised to a reference measurement of the empty sample cell. The peak-to-peak amplitude of the time domain waveform is then calculated by dividing the peak-to-peak value of the sample by the peak-to-peak value of the reference measurement (both shown for a typical THz spectrum in Fig. 3).

As presented in the schematic of Fig. 1, wafers are highly porous structures with many of their pores of the order of hundreds of micrometers. Focused terahertz radiation, with wavelengths ranging from 3 mm at 0.1 THz to 100 μm at 3 THz, will scatter strongly from pores at sizes of the length scale of the wavelength. To average out local pore inhomogeneities the wafer samples are placed off focus in the beam path instead of at the focal point. The results in a transmission measurement averaged over a larger volume of the sample. In our case the beam is estimated to penetrate a cross section of 6 mm in diameter on the surface of the wafer.

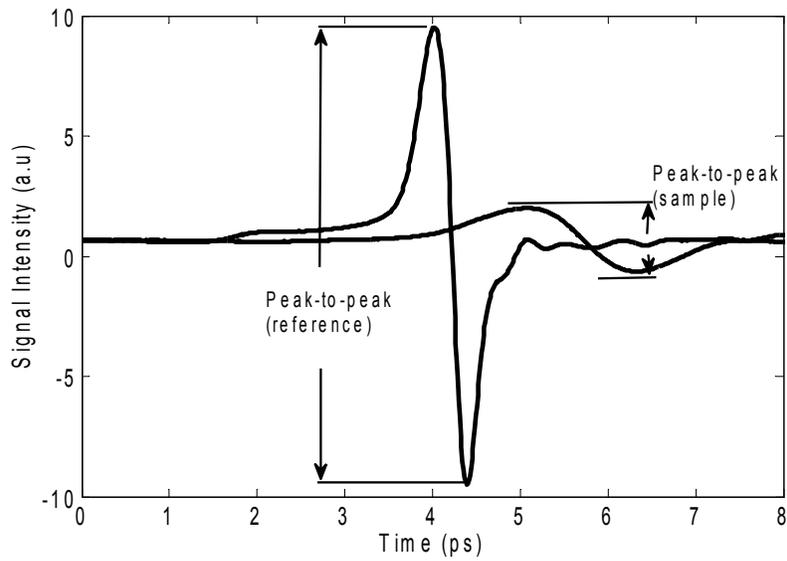


Fig. 3 Time domain THz waveforms of the reference and the sample (typical). The peak-to-peak amplitude of the sample is normalised to that of the reference.

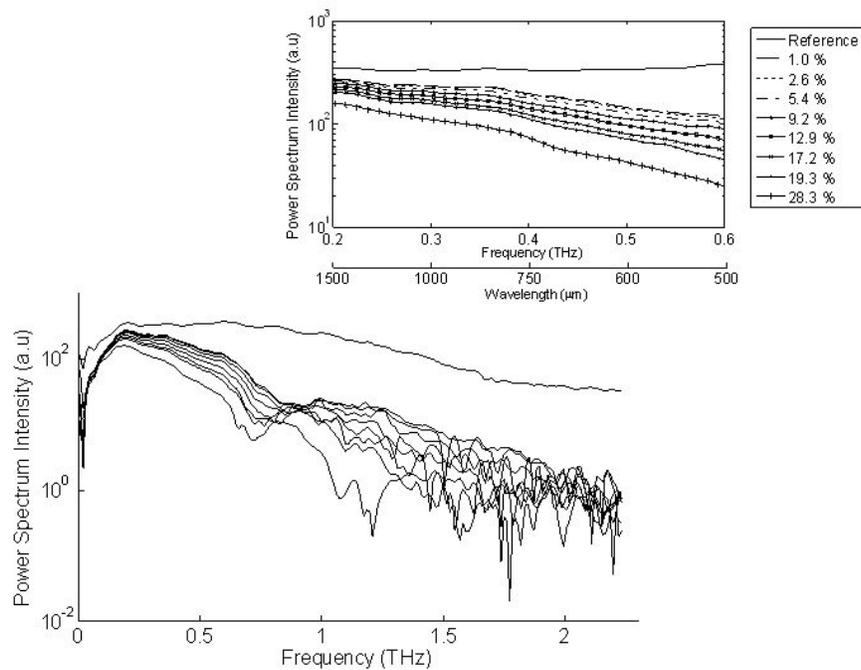


Fig. 4 Spectra of samples with known moisture content. In the inset the region between 0.2 and 0.6 THz is magnified.

3. Results and Discussion

The optical density spectra of samples of Type I wafer at different moisture content (as

determined gravimetrically) are shown in Fig. 4. The amplitude in the lower frequency region exhibits a direct relation to the moisture content, this can be more easily seen in the extracted inset. As the moisture content increases, the samples absorb more terahertz radiation and the spectra decrease in amplitude. In the region between 0.2 and 0.6 THz, the spectra are well defined and separated for each individual sample. For frequencies above approximately 0.6 THz the wavelength decreases to less than 0.5 mm which is the size of the biggest pores and hence scattering becomes the major contribution to the loss of terahertz radiation rather than absorption from the water content. Hence, at these higher frequencies the amplitude is more sample dependent and cannot be used to accurately measure the moisture content.

As described in the previous section, the normalised peak-to-peak amplitude of the time domain waveform was easily measured. For the sets of wafer samples of Type I and II with known moisture content (determined gravimetrically), the peak-to-peak amplitude is plotted against the moisture content and shown in Fig. 5. As the moisture content increases the peak-to-peak amplitude decreases due to the absorption by the water content.

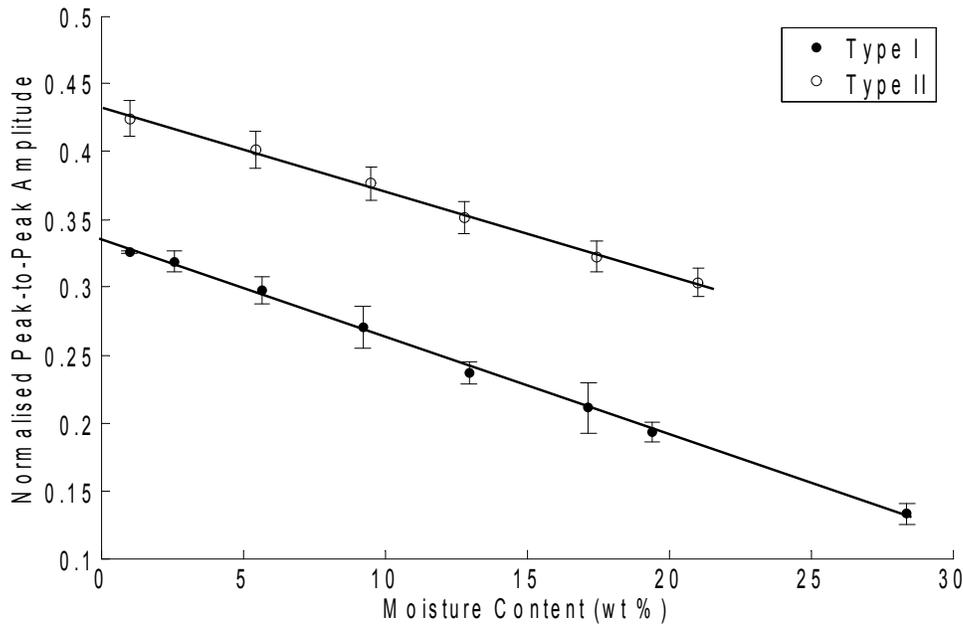


Fig. 5 Calibration curves of the moisture content against the normalised peak-to-peak signal intensity of both Type I wafer, and Type II wafer. The solid lines represent the best fits obtained with linear regression ($R^2 = 0.9985$ and 0.9991 for Type I and II respectively).

Both types produce very linear dependencies, the best fits obtained with linear regression are $y = -0.0062x + 0.43$ ($R^2 = 0.9985$) and $y = -0.0072x + 0.34$ ($R^2 = 0.9991$) respectively. y is the normalised peak-to-peak amplitude whilst x is the moisture content. The standard errors are $3.2E-5$ for Type I wafer and $5.8E-5$ for Type II wafer. Visually a plot of the residual revealed no systematic bias as a function of moisture content. A two tail t-test was performed to check for zero residual mean and hence the absence of bias. This revealed no bias at the 95 % confidence interval. Calibration curves can thus easily be obtained directly from such time-domain data. The origin of the error bars (which result predominately from laser fluctuations) is taken as the standard deviation of the mean of the scans that were co-added to produce each waveform. With

reference to Fig. 4, in excess of 96 % of the signal is detected between the lower frequencies (0.2 – 0.6 THz), as determined by summation either side of 0.6 THz. Thus the time domain signal is dominated by this 0.2 - 0.6 THz contribution. By comparing the two sets of data in Fig. 5, it is immediately obvious that Type I wafer samples have a lower THz transmittance relative to Type II wafer. This is consistent with the lower porosity of Type I wafer, and hence greater total moisture content encountered by the THz beam. Appropriate analysis of data acquired with both the moisture analyser and the THz device revealed an accuracy of 0.3 wt % (which is comparable to that of the moisture analyser) and a precision of 0.5 wt % (although this improves significantly as more data is co-added to improve the signal-to-noise ratio).

In the second experiment dry Type I wafer sample was exposed to humid air and then placed in the flow-cell where dry nitrogen was continuously blown over the sample, thereby drying it. The initial moisture content of the wafer sample was determined gravimetrically at 11.8 wt%. A time series of THz transmission measurements was then acquired. The evolution of the peak-to-peak amplitude signal together with the extrapolated moisture content from the calibration curve obtained from Fig. 5 is shown in Fig. 6. At the end of the experiment the actual moisture content of the sample was measured gravimetrically using the moisture analyser and was found to be 3.8 wt%, in excellent agreement with the THz transmission measurement as shown.

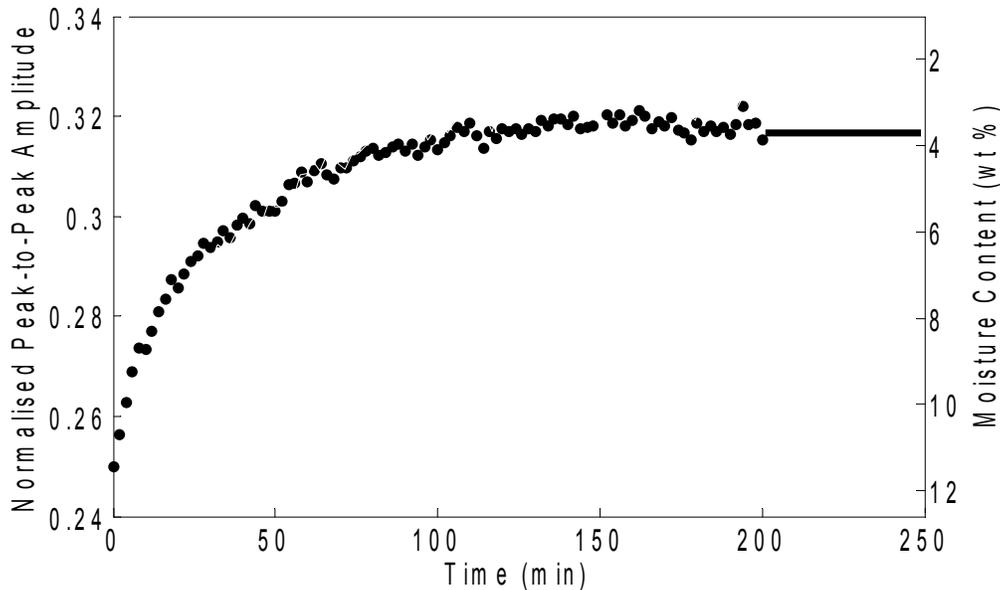


Fig. 6 Time series of transmission measurements for the drying experiment. The extrapolated (from calibration data) moisture contents are also shown. Agreement with the gravimetric measurement on the final sample (thick solid line) is excellent.

As seen from Fig. 4, and discussed earlier, the effect of moisture content is more pronounced at lower terahertz frequencies. As a consequence, there is the possibility of using a potentially cheaper, fixed frequency continuous wave terahertz system to measure moisture content. The hardware for fixed frequency sub-terahertz measurements is an area of intense research activity and the modern systems are compact and suitable for online applications [32, 33]. Potential benefits of such systems also include a better signal to noise ratio, hence the method will be more suitable for on-line applications. The potential for a system operating at fixed frequencies is now

explored using the data from Fig. 4. The power spectrum intensity at one frequency (in this case at 0.4 THz) is plotted against moisture content in Fig. 7.

The relationship between amplitude and moisture content is again linear; the best fit obtained with linear regression is $y = -0.015x + 0.53$ ($R^2 = 0.9988$) where y is normalised spectral intensity and x is moisture content. The standard errors are $3.2E-5$ and a two tail t-test revealed no bias in the residual at a 95 % confidence interval.

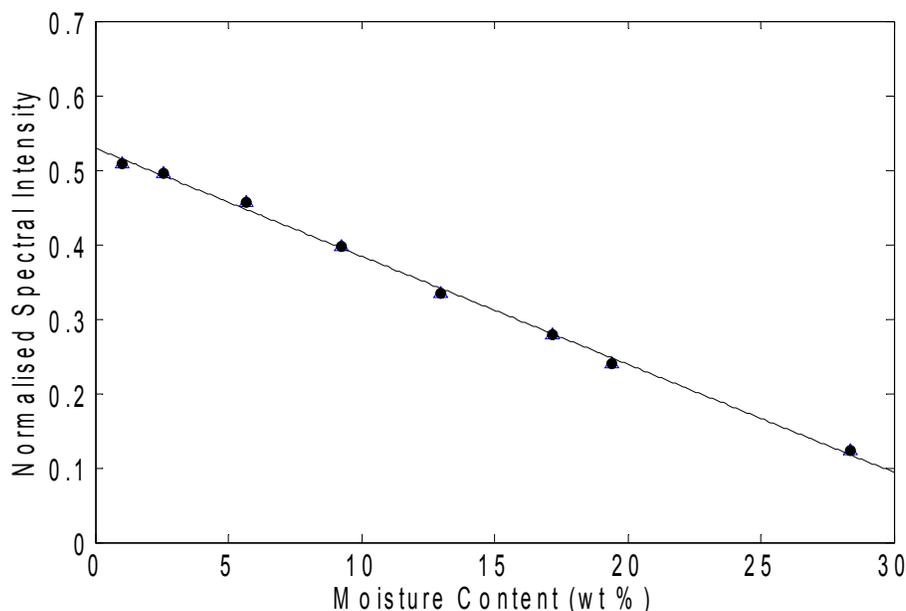


Fig. 7 Normalised spectral intensity at 0.4 THz. The line represents the best fits obtained with linear regression ($R^2 = 0.9988$).

4. Conclusion

In this work a non-invasive quantitative method for measuring the moisture content of food wafers has been explored using terahertz radiation. The selective absorption of this part of the electromagnetic spectrum by water shows potential for a very accurate non-invasive method that requires very simple calibration against a standard gravimetric method. Application of fixed frequency systems could lead to very rapid, compact and accurate on-line measurement.

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