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Method and Instrument for Measuring Local Water Content Inside Food

Karin Thorvaldsson & Christina Skjöldebrand

SIK, The Swedish Institute for Food Research, Box 5401, S-402 29 Göteborg, Sweden

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ABSTRACT

A new method of measuring local water content inside food during heat processing has been evaluated. The method uses a fibre optic NIR-Instrument. The evaluation was mainly based on investigations of the influence of structure and temperature. The instrument was found to be very sensitive to both the structure and the temperature of the sample. The dependence on the temperature of the sample can be incorporated into the calibration and is thereby not an impediment to the measurements. The structural dependence makes quantitative measurements at different places in the sample difficult for inhomogeneous materials. However, measurements of dynamic changes in the water content in one spot work well for both homogenous and inhomogeneous materials and are probably the best application of the instrument. By using two or more optic fibres, the diffusion of water can also be measured. Copyright © 1996 Elsevier Science Limited.

INTRODUCTION

Background

When food is heated, water transport starts inside the food and the water content distribution changes. During the heating process, physical and chemical changes take place inside the food, e.g. protein denaturates, starch gelatinises and aroma components are formed. These changes are strongly affected by the water content. The final taste and structure in different parts of the food is thereby affected by the water transport. A good example of this is the difference in taste and structure between the crust and the crumb of a bread loaf. It is therefore of great importance to be able to measure and predict the water content distribution and the water transport during heating processes.

Measuring methods

Possible methods of measuring the water content distribution in food products are near infrared reflectance (NIR) in combination with fibre optics, nuclear magnetic resonance imaging (NMR) and computer tomography (CT). NMR and CT give cross sections of the water distribution in the sample material (Webb, 1988; Attard, *et al.*, 1991; Morris, 1986), but it is often difficult to imitate the conditions of the heating process and to measure the temperature. Metallic details like thermocouples disturb the imaging. NIR with fibre optics can be used to measure the water content in small, well defined spots inside the sample. The benefits of this method are twofold. Firstly, the instrument can be used in the process plant, since only the optic fibre has to be placed in the heating equipment. Secondly, the temperature can be measured with ordinary thermocouples without disturbing the measurements. The method of NIR with fibre optics is described and evaluated in this paper.

DESCRIPTION OF THE METHOD AND INSTRUMENT

Theory of method

All molecules have specific resonance frequencies, corresponding to bending and vibration motions in the molecule. When light with any of these resonance frequencies is sent towards the molecule, it is absorbed and transformed into kinetic energy. All types of molecules have different resonance frequencies which give them specific absorption spectra. The water molecule has absorption peaks in the near infrared region at 0·97, 1·19, 1·45, 1·94 and 2·95 μm.

To measure the local water content of a sample, light is sent into the sample through an optic fibre and the intensity of the reflected light is measured. The absorption peaks at 1·45 and 1·94 μm are best suited for these measurements. For the two shorter wavelengths, the absorption peaks are too weak to give a good signal–noise ratio and for the longer wavelength the absorption is so effective that the intensity of the reflected light becomes too weak. The weaker peak at 1·45 μm is therefore best suited for high water contents, while the stronger peak at 1·94 μm is used for low water contents.

Temperature dependence

The absorption peak used in this study is the one at 1·45 μm. This wavelength corresponds to the first overtone, i.e. the double frequency, of the 0–H stretch in the water molecule (von Hippel, 1988; Williams, 1984). When the temperature rises, the absorption peak shifts towards shorter wavelengths (see Fig. 1, Rodgers, 1981). In liquid water, three or four molecules often bond together with hydrogen bonds and when the temperature rises some of these bonds are untied (Rodgers, 1981). The resonance frequency of the 0–H stretch differs for a molecule with hydrogen bond and for a molecule without a hydrogen bond. The absorption peak at 1·45 μm is a superposition of these two peaks. At the wavelength 1·441 μm, the absorption is almost constant for the temperature range 20–100°C (see Fig. 1; Rodgers, 1981), therefore this is the optimal wavelength to use.

Instrumental design

Figure 2 shows a schematic picture of the instrument. Light from an infrared light source (1) is collected and directed by a lens (2) and sent through a rotating filter wheel (3). The filter wheel contains at least three filters which select one wavelength each: one main wavelength, at the peak, and two reference wavelengths, one on each side of the peak. The filter wheel is synchronized with the detector (8). After the filter wheel, the light is collected by another lens (4) and focused into the optic fibre (5). The optic fibre ends in an optic probe (6) that is placed in the sample. The probe comprises a lens, which directs the light into the sample and collects the reflected light and sends it back through a receiving fibre (7). The receiving fibre leads the light to the detector, where the intensity is measured. After the detector, the signal goes to an analysing unit (9), where the intensities from the different wavelengths are weighed to get the water content.

The fibre optic probe is constructed, as shown in Fig. 3, with a graded index lens (11). The lens is cut at an angle (10), where the reflected light is out of the critical angle of the lens. This means that the directly reflected light, i.e. the light that has not entered the sample, is not measured. The probe is also made of dielectric material (12) for use in electromagnetic fields, like microwave ovens. The probe is 3 mm in diameter and the measured sample volume is 2–3 mm^3, with small variations due to the structure of the sample. The volume (13) being measured is in front of the probe, so as not to disturb the transport of water through the volume.

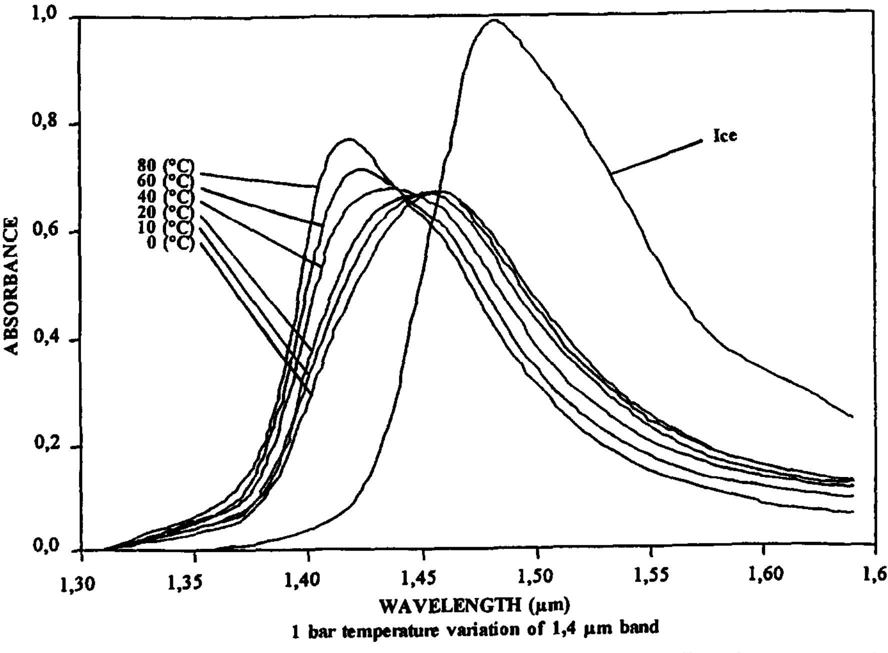

Fig. 1. Temperature dependence of the absorption peak for liquid water at 1·45 µm. (Rodgers, 1981).

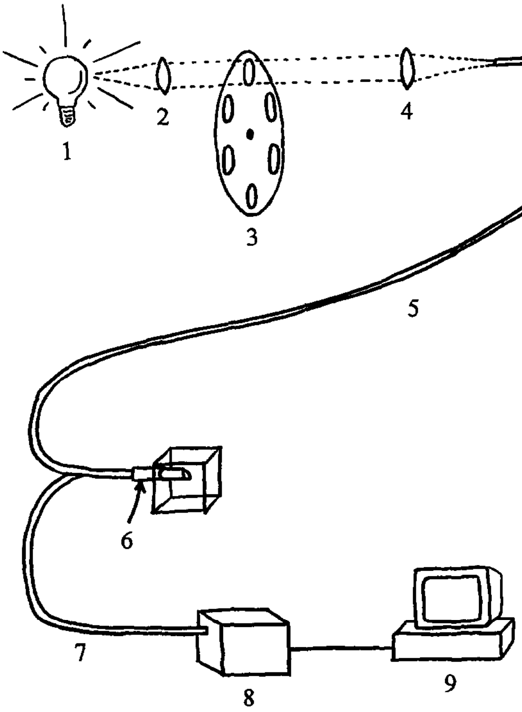

Fig. 2. Schematic view of the fibre optic water content measuring instrument.

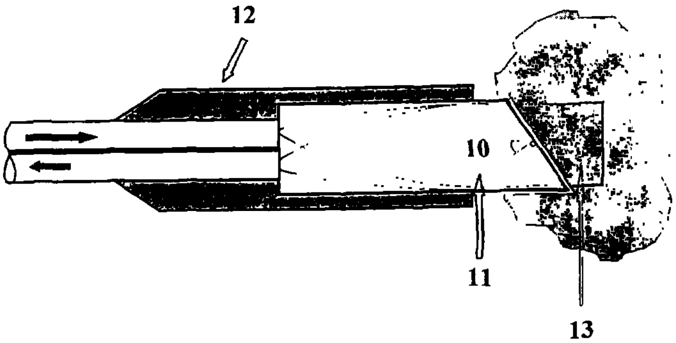

Fig. 3. The fibre optic probe.

Calibration of the instrument

The instrument has to be specially calibrated for each food material that is measured.

Not only the water in the food absorbs light, the food itself also absorbs light. The total absorption is measured at three wavelengths, one at the absorption peak of water and one on each side of the peak, as described above. The intensity of the absorption at the centre of the peak is divided by the weighed sum of the intensities of the other two.

$$S=\frac{I_{peak}}{a^{*}I_1+b^{*}I_2} \tag{1}$$

where $a+b\times 1$, S is a measure of the water content, but it has to be related to the sample material. The two reference wavelengths compensate for disturbances that affect the total absorption intensity, like variations on the main voltage, sunlight at the sample etc. The variables *a* and *b* are determined out of the absorption by the food itself so that $aI_1=bI_2$. The absorption varies linearly with the water content (Verbiese, 1981). By measuring S for the dry sample, S_{W0}, and S_{W1}, for a sample of known water content, W_1, a calibration equation is obtained:

$$W=\frac{W_1}{S_{W_1}-S_{W_0}}(S-S_{W_0}) \tag{2}$$

where W is the water content. S_{W0} and $W_1(S_{W1}-S_{W0})$ are constants, specific for each material. The equation can be rewritten as

$$W=c_0+c_1{}^{*}S \tag{3}$$

where c_0 and c_1 depend on the structure and composition of the material being measured.

EVALUATION OF THE METHOD AND INSTRUMENT

Structural dependence

The effect of the structure of the material was studied, by changing the position of the probe in the material. White bread from three different loaves was used as reference material in all the experiments. All the bread was prepared under the same conditions and baked from the same recipe (for 7 loaves):

1800 g	wheat flour
30 g	salt
30 g	sugar
30 g	margarine
100 g	yeast
1000 g	water

The dough was risen twice and the loaves were baked at 220°C. After baking, the bread was left to cool down, then the crust was cut away and the crumb was placed in a plastic bag to rest for some hours to ensure a uniform water distribution. After the rest, a piece of bread was weighed and dried in an oven at 105°C for several hours to determine the water content. The water content of the bread was found to be 41%. Another piece of the bread was used to measure the water content by the fibre optic instrument. The optic probe was placed at several different sites in the bread and the water content was read from the instrument.

Temperature dependence

The temperature dependence of the absorption wavelength in the bread was evaluated. The same type of white bread as in the structural studies was used. The water content was determined by weighing and drying. A 3 mm thick squared slice with 50 mm sides was cut out from the bread. The slice was placed between two metallic plates with 200 mm sides and with 3 mm compact waterproof isolation in between, except for a square in the centre, where the bread slice fitted (see Fig. 4). The fibre optic probe and a thermocouple were placed in the centre of the bread slice. The metallic plates were pressed together with clips. The plates, with the bread and the sensors, were then placed in an oven at 180°C and heated until the bread reached 90°C and then taken out of the oven to cool down to 30°C. This procedure was repeated 3–4 times. After the last cooling down, the bread slice was taken out of the metallic plates and the water content was determined by weighing and drying, to see that the water content was still the same as before the experiment. Since the temperature dependence is influenced by the water content of the sample, dry bread was also investigated by the same method. Samples from three different loaves were used for the experiments.

RESULTS OF THE EVALUATION

Structural dependence

Figure 5 shows a typical diagram of the measured water content vs time, where the probe has been moved from one site to another every fourth to fifth minute. The

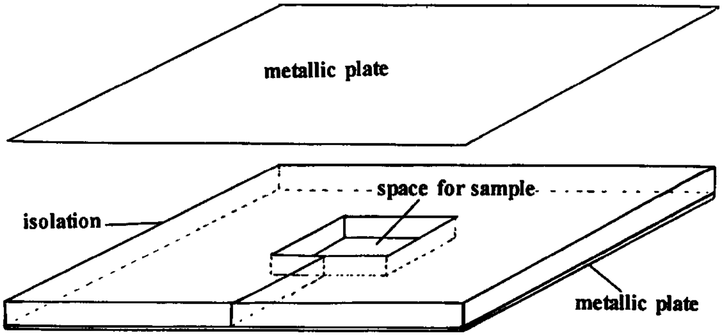

Fig. 4. The design of the metallic plates for measuring the temperature dependence of the material.

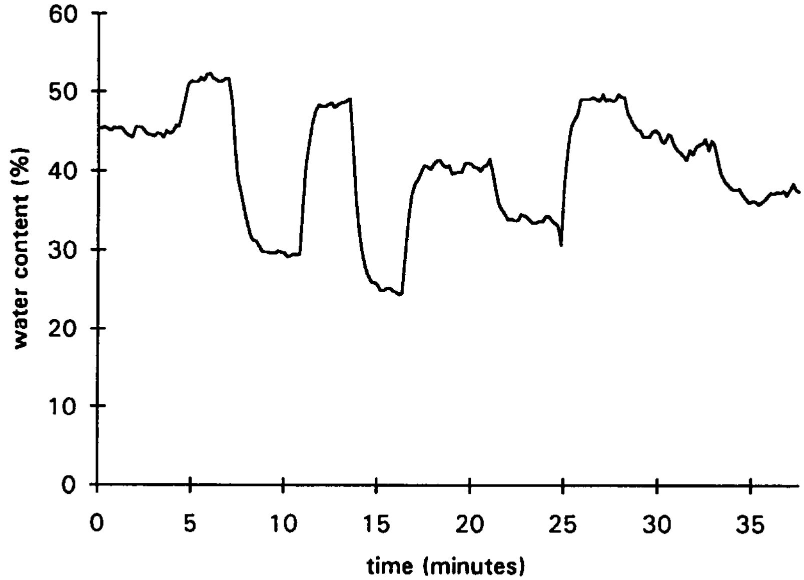

Fig. 5. The measured water content at different probe sites. The probe was moved to a new site every 4–5 min.

diagram shows a strong dependence on the probe site. In Tables 1 and 2, the water content at the different sites is listed and the average water content with standard deviation is calculated for the three samples. The average water content and standard deviation was 40 ± 8·3%.

Temperature dependence

The instrument also showed a strong dependence on the sample temperature (see Fig. 6). When the temperature of the samples was increased from 30 to 90°C, the measured water contents rose about 0·16 ± 0·02 and 0·11 ± 0·01%/°C for the moist and dry samples, respectively (see Table 1), while the true water content was kept

TABLE 1
Structural Dependence: Measured Water Content at Different Probe Sites

Probe site	*Sample a:*	*Sample b:*	*Sample c:*
1	48	39	44
2	30	33	51
3	39	48	30
4	46	43	48
5	36	37	29
Average:	39·8 ± 7·4	40·0 ± 5·7	40·4 ± 10·3

TABLE 2

Temperature Dependence: Measured Change in Water Content for the Moist Samples Due to Temperature (%/°C)

Nr.	*Sample a:*		*Sample b:*		*Sample b:*	
	Rise	*Sink*	*Rise*	*Sink*	*Rise*	*Sink*
1	0·22	0·16	0·16	0·12	0·16	0·12
2	0·16	0·17	0·15	0·18	0·16	0·15
3	0·15	0·17	0·15	0·17	0·13	0·13
4	0·18	0·17	0·19	0·18	0·15	0·17
Average:	0·17 ± 0·02		0·16 ± 0·02		0·15 ± 0·02	

almost constant. The true water content at the start was 40·6 ± 0·3% and after the measurements it was 39·7 ± 0·1%. The first temperature peak gave rise to a stronger temperature dependence than the other peaks.

DISCUSSION

Structural dependence

The results show that the instrument is very sensitive to where the probe is placed. This is probably due to the structure of the sample material in front of the probe. Since the volume being measured is so small, the site of the probe is crucial in

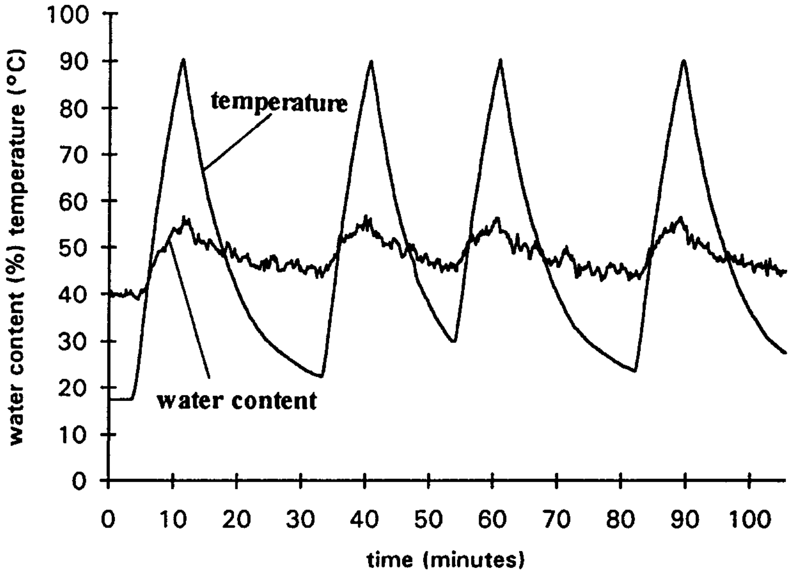

Fig. 6. The measured water content at varying temperatures.

determining how the light is reflected. The measured signal is strongly affected by the position of the probe either in a bread pore or at a pore wall. However, if a hole for the probe is first prepared with a drill, so that the probe looks at a crumbly surface, then almost the same water content can be read from the instrument for the different probe sites (see Fig. 7).

Temperature dependence

The results also show that the measured water content is strongly affected by the sample temperature. As shown in Fig. 1, the absorption peak of water shifts towards shorter wavelengths with increasing temperature. The same mechanism probably takes place for the other molecules in the bread, though some of them could shift towards shorter and some of them towards longer wavelengths. The total absorption will be a superposition of all the absorption peaks.

Another contribution to the temperature dependence of the instrument is the fact that only the liquid water is measured. In the samples there could also be a gas phase with water vapour. This is true for most foods and especially for bread, and the equilibrium state between the liquid and the gas phase depends on temperature.

However, both these effects and probably some others will be included in the measured temperature dependence (see Fig. 6). To obtain the true water content, W_t, the measured water content, W_m, has to be corrected in accordance with the measured temperature dependence, Td.

$$W_m = W_t + (T - T_S)^* \mathrm{Td} \tag{4}$$

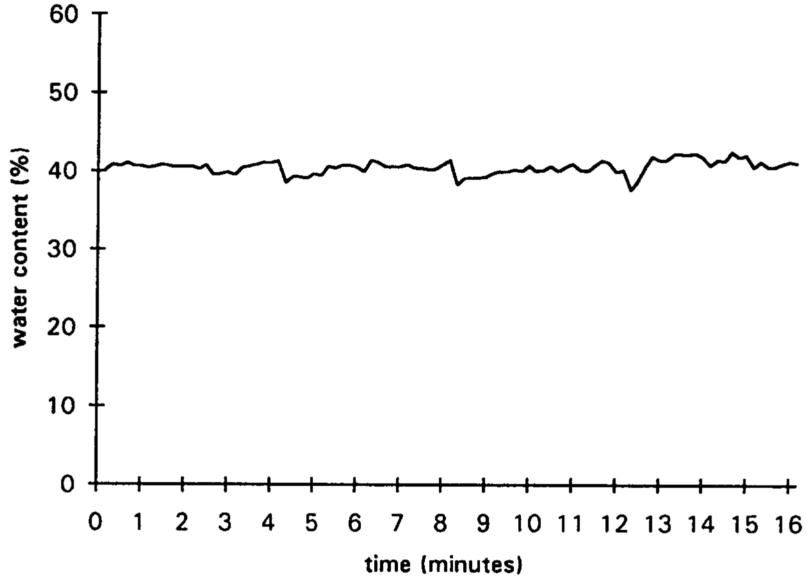

Fig. 7. The measured water content at different probe sites, with pre-drilled holes for the probe. The probe was moved to a new hole with 4–5 min intervals.

where T and T_S are the temperature and the initial temperature, in °C, respectively and Td is

$$Td=Td_{dry}+(Td_{moist}-Td_{dry})\frac{W_t}{W_s} \tag{5}$$

where Td_{dry} and Td_{moist} are the temperature dependencies for the dry respective moist samples in %/°C and W_8 is the initial water content in %. By combining eqns (4) and (5) W_m can be written as

$$W_m=W_t+(T-T_s)^*\left(Td_{dry}+(Td_{moist}-Td_{dry})\frac{W_t}{W_s}\right) \tag{6}$$

which gives

$$W_t=\frac{W_m-(T-T_s)^*Td_{dry}}{W_s+(T-T_s)^*(Td_{moist}-Td_{dry})}{}^*W_s \tag{7}$$

The correction described above is only valid when the instrument is calibrated for the studied material at the initial temperature and water content.

CONCLUSIONS

The fibre optic instrument is well suited for dynamic studies where the probe is placed at the same site during the whole measurement. The instrument then has to be calibrated for the light absorption by the material and for the temperature dependence on the absorption spectra. Drying and heating are examples of processes where the instrument works well.

The strong structural dependence of the instrument makes quantitative measurements difficult. However, if the structure is very homogenous, even quantitative measurements can be good.

The most useful application of the instrument is probably to measure water transport. This is achieved by using two or more optic probes placed at different sites in the sample. The possibility of measuring the combined heat and water transport will advance food engineering research by enabling us to understand, predict and control heat processes.

Desirable changes that could improve the instrument are that (1) the light source could be exchanged for a diode laser to improve the intensity and thereby the stability of the signal. However, the light must not be so intense that it heats the sample. (2) The optic probe could be equipped with some thin material at the top. This material should be able to quickly adjust its water content to the water content of the surrounding material. Thereby the instrument would not have to be calibrated, since it would always measure the same material, volume and structure.

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