A method of determining the quantity of unbound water in a substrate is disclosed, comprising directing a beam of incident microwave radiation of a wavelength absorbable by the unbound water; measuring the microwave radiation transmitted through the substrate, and determining the quantity of water present from the intensity difference between the incident radiation and the transmitted radiation. This detection method allows such processes as hydration reactions, dehydration reactions, freezing and drying to be monitored as to their progress. A suitable frequency of incident microwave energy used is 3 GHz or greater, preferably in the range of 3 to 30 GHz, more preferably in the range 9 to 13 GHz, yet still more preferably within the range 10 to 12 GHz. A particularly preferred range is between 10 and 11 GHz. The application also describes compensation for dissipation, absorption etc. by superimposing a low frequency reference signal. The received carrier wave strength can then be compared with the received reference wave strength in order to calculate the carrier wave loss through water absorption. A preferred frequency for the reference signal is not greater than 100 kHz, more preferably between 10 Hz and 10 kHz. A suitable frequency within this range is 1 kHz. A less preferred (but viable) frequency is 81 Hz. The invention also provides apparatus embodying the above methods.
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APPARATUS FOR MEASURING THE WATER CONTENT WITH MICROWAVES

This invention relates to apparatus and a method for determining the quantities of free water in a substrate or other sample (hereinafter "substrate").

There are numerous circumstances in which it is most desirable to be able to determine the residual levels of water in a substrate, for example on drying solid. For example, in the production of cellulose or wood pulp based products such as chipboard, pulp board, plywood, paper and cardboard, it is often desirable to know the residual levels of water present in the material at a given stage in its processing. Similarly, in relation to insulation and building boards, calcium silicate boards, and concrete and cement precast sections, it is often useful to know the residual concentrations of water present in the material. Additionally, in a food freezing plant it is desirable to know the liquid water content as opposed to the frozen water content. In gas phases, the invention can measure the gaseous level of moisture from which a relative humidity value can be derived. The invention also allows a reaction which proceeds by condensation or elimination of water to be followed, eg the manufacture of nylon, terylene, urea formaldehyde etc. It is also useful for determining the progress of the hydration of gypsum, both synthetic and natural, and all hydrate forms (ie anhydrous and hemihydrate). It allows the end point of the rehydration reaction to be determined
accurately and swiftly, and in preferred forms is able to give information regarding the maximum rate of rehydration. Other circumstances exist which will be exemplified later.

The present invention provides a versatile and simple method of determining the amounts of fluid water in such substrates.

The present invention makes use of a microwave emitter and receiver to determine the amount of liquid water by inference from the levels of microwave radiation absorbed by free water in the substrate. The level of bound water can then be calculated if required.

Microwaves are electromagnetic waves which have a wavelength between the wavelengths of infrared and radio waves, typically in the region of about $1 \times 10^2$ metres, and a typical frequency in the region of $3 \times 10^{10}$ Hertz.

A rotating molecule can withdraw energy from electromagnetic radiation or give up energy to the radiation if it can interact with the oscillating electric field associated with the radiation. A molecule can do this if it has a dipole moment. A rotating dipole provides a coupling with the oscillating electric field of the radiation and allows energy to be transferred from the radiation to the molecule, or vice-versa. The rotational spectra of most molecules occur in the microwave spectral region.

According to the present invention, there is provided a method of determining the quantity of unbound water in a substrate, the method comprising directing a beam of incident microwave radiation of a wavelength absorbable by the unbound water; measuring the microwave radiation transmitted through the substrate, and determining the quantity of water present from the intensity difference between the incident radiation and the transmitted radiation.
The inventor has found that bound water, for example that in ice or that present as water of crystallization does not attenuate microwave energy. This is because such water is not free to rotate. Therefore, as free water is removed from a substrate, for example by uptake during a hydration reaction, by freezing, or by bulk loss through drying, the variation in free water content can be followed through the attenuation rate of incident microwave energy.

This detection method allows such processes as hydration reactions, dehydration reactions, freezing and drying to be monitored as to their progress.

Preferably, the frequency of incident microwave energy used is 3 GHz or greater. More preferably, the frequency used lies through the range of 3 to 30 GHz, more preferably in the range 9 to 13 Gzh, yet still more preferably within the range 10 to 12 GHz. A particularly preferred range is between 10 and 11 GHz.

The power of the incident microwave energy need not be great provided that it is sufficient to penetrate the sample depth. Excessive microwave power may cause a heating effect which could affect the water content being measured. It is therefore preferred if the incident power is less than 20 mW, more preferably less than 15 mW. A suitable strength is 8mW.

It is also possible to measure the incident power in terms of the energy arriving at the substrate surface. In that case, it is preferred if the incident energy is less than 0.6 Wcm².

There will inevitably be some loss of microwave energy through scattering, refraction and other reasons unconnected with water content. In a preferred form of the invention, therefore, the previously described
incident microwave energy, hereinafter referred to as a "carrier wave", is transmitted together with a superimposed low frequency reference signal. The frequency of this reference signal is not critical to the invention, so long as it is not within a frequency range absorbable by free water or the substrate. In this preferred arrangement, the received carrier wave strength can be compared with the received reference wave strength in order to calculate the carrier wave loss through water absorption.

A preferred frequency for the reference signal is not greater than 100 kHz. A more preferred range is between 10 Hz and 10 KHz. A suitable frequency within this range is 1kHz. A less preferred (but viable) frequency is 81 Hz.

The invention also provides apparatus for determining the level of unbound water in a substrate, the apparatus comprising a microwave transmitter; a microwave receiver; means for comparing the relative intensities of the incident radiation and transmitted radiation, and converting the difference into a displayed value indicative of water content; and a display for displaying the said value.

Preferably, the microwave transmitter is adapted to emit a carrier signal and a superimposed reference signal, the reference signal being of a frequency not substantially absorbable by either water or the substrate, the means for comparing the relative intensities being adapted to compensate for signal dissipation through use of the reference signal strength.

Embodiments of the invention will now be described by way of example with reference to the accompanying drawings, in which;

Figure 1 is a schematic illustration showing the components of the microwave detection apparatus according to the first embodiment of the invention;
Figure 2 is a plot calculated using an embodiment of this invention showing the progress of a hydration reaction by plotting the levels of residual free water in the substrate;

Figure 3 is a schematic representation illustrating the effect of the reference signal;

Figure 4 is a schematic illustration of a second embodiment of the present invention; and

Figure 5 to 10 are graphs showing the variation in output of an embodiment of the invention over time in several situations.

Referring to Figure 1, there is a substrate which is under consideration. The substrate 10 comprises, for example, a concrete sample undergoing a hydration reaction.

A frequency generator 12 generates a microwave carrier signal at approximately 10.4 GHz, on which is superimposed a 1 kHz reference signal. This is fed to a transmitter 14, which emits an 8mW electromagnetic signal represented as 16. This signal is directed toward and through the sample 11, and is detected by a receiver unit 18 which passes the receiver signal to a demodulator and calculation unit 20.

The calculation unit 20 compares the relative signal strengths of the reference and carrier waves. Loss of signal strength in the reference wave is assumed to relate to dissipation and scattering losses and hence any further loss beyond this level in the carrier wave can be traced to absorption by free water in the sample. This loss is quantified and output as a readable result. The output may be in the form of a digital display or a computer-readable electronic signal.
In a variant of the above embodiment shown in fig 4, the transmitter and receiver are both on the same side of the sample, and a reflector is positioned behind the sample so that the electromagnetic signal is reflected back, passing through the sample twice. The reflector is suitably of a metallic material. Some samples to which this invention can be applied are supplied with a metal backing or in a metal casing, in which instance such backing or casing can serve as a reflector. This enables the invention to be employed without the need to gain regular access to the rear of the sample.

Figure 2 shows results achieved from a prototype being an embodiment of the invention. The sample was a 6 mm thick gypsum cell, and a 10.4 GHz signal was used. The horizontal axis shows the time in minutes after preparation of the gypsum slurry, whilst the vertical axis shows the attenuation in decibels (dB). It can be seen that the attenuation is initially high, indicating a strong signal loss but this attenuation eventually settles down to a constant smaller figure as the hydration reaction completes.

Two samples are shown, labelled A and B. The absolute values of attenuation vary between the A and B samples, but this can be related to differences in the preparation of the gypsum samples and differences in the substrate sizes and geometries. It is relevant that the ratio of the initial and final attenuations is comparable between the two samples. The present invention therefore enables the progress of the hydration reaction to be followed and the point of completion of the reaction to be accurately and speedily determined.

Figure 3 illustrates the use of the reference signal. The vertical axis shows signal strength, and the transmitted carrier and reference signals are designated as Tx_C and Tx_R respectively. The carrier and reference signals as received are designated by Rx_C and Rx_R respectively.
For ease of explanation, it is assumed that the signal strengths of the two transmitted signals $T_X$ and $T_R$ are identical. This is not necessarily the case in practice, but can easily be compensated for. The received reference signal has been attenuated by a certain amount due to scattering etc. and this is indicated as the signal strength drop $A_R$. Using this information, the attenuation due to water ($A_W$) can be calculated from the signal strengths of the carrier and reference waves $S_C$ and $S_R$ respectively as

$$A_W = \frac{S_R - S_C}{S_R}$$

The apparatus and method set forth in this Application is applicable to a wide variety of industrial applications. Essentially, any application where it is desired to measure the free water content without detecting bound or lost water is capable of benefitting from this invention. For example, a frozen food manufacturer could use the system to ensure a product is thoroughly frozen to the necessary quality. Paper manufacturers can monitor the transition from pulp to paper. Laminating and sheet timber products can use the system to measure the take-up of water in the glue component and hence detect when the product is set hard. Pre-cast concrete producers and purchasers can use the invention to check on the stage of reaction in concrete, e.g. as delivered. This would distinguish between fresh concrete and part-reacted concrete to which water had been added to render the slurry to the appropriate viscosity. This latter practice produces concrete with a very low bulk strength and a reliable detection means such as that set forth in the invention is thus able to increase confidence in a product as supplied. The invention is also applicable to measurement of water in hydrolytic mortars such as silicious cement and lime mortars. Reactions which proceed by condensation or elimination of water can be tracked. Commercially important examples of such reactions are the production of nylon and terylene. In the agricultural sector, sugar beet or grain etc. can be tested using the invention and sorted according to the water content. Grain which is dry can be put immediately into storage
without unnecessary drying. Suppliers of sand, ceramics, etc. can use the invention to calculate the true weight of product delivered (i.e. less free water). Industries which require the application of a wet surface coating can use the invention as a simple depth testing gauge and hence coating weight reading. Manufacturers of brick and clay products, for example bricks, can use the invention to ascertain the level of drying in products being produced. The meat industry can use the invention as a simple lean/fat ratio detector, based on the fact that lean meat is damp whilst fat contains essentially no water. Manufacturers of flocculants, colloids and gels can use the invention to measure the percentage of water taken up by the flocculant. Such water is essentially bound and not free. Products which have recently been centrifuged or filtered can be tested to ascertain the amount of water removed from the caked product. The invention can also be used as a humidity measurement device, for example across air-conditioning or other gas supply ducts, or to distinguish between gaseous water and water vapour. The manufacture of gypsum proceeds via a rehydration reaction, which can be followed via the present invention to avoid unnecessary waiting.

It will therefore be clear that the present invention has a wide variety of applications. Essentially, any non-metallic substrate containing free water can be analysed for the water level. Examples from several such situations are shown in Figures 5 to 10.

In Figures 5 to 10, the embodiment used involved a 10.4 GHz signal upon which was superimposed an 1 kilohertz reference signal. The total signal strength was 8 mW, or 0.6 Wcm⁻² expressed as energy arriving at the substrate surface.

Figure 5 shows an inferred plot of bound water vs time for a sample of ice which is allowed to thaw from -15°C towards zero. The attenuation due to free water is measured; in a closed system such as that monitored
any increase in the free water content must be due to a decrease in bound water. Thus, an inverse plot of free water, as in Figure 5, gives a clear representation of bound water.

As time progresses, the amount of free molecular water gradually increases. It appears that a phase change or recrystallisation step takes place at 1084 seconds. The bound water content then declines relatively steadily towards a minimum value.

The invention is therefore clearly able to distinguish ice from free water. Once a particular physical arrangement has been characterised fully, data such as shown in Figure 5 can be used in a straightforward manner to calibrate the signal in terms of the relative proportion of ice and free water.

Figure 6 shows the results obtained from a system set up to monitor the output of a laminating machine. Clearly, if a small area of the output sample is measured then the amount of water will be directly proportional to the thickness of the glue sample. Such a measurement is of use in controlling the glue thickness so as to achieve a minimum level thereof, which is consistent with adequate performance but is not excessive. An excessive glue thickness could lead to lesser performance and will inevitably lead to waste.

In the test conditions shown in Figure 6, the glue application rate was manually adjusted at three minutes and four minutes from the start of the test. This adjustment is clearly visible in the output results. The present invention therefore allows a feedback to be established in order to control the glue application rate.

Figure 7 shows the results of a calibration step for the arrangement of figure 6. The data is averaged over a short time period in order to eliminate the process noise shown in figure 6. A suitable time period is
approximately equal to the period of the noise spikes, i.e. about 5 seconds. The glue applicator was manually adjusted and the application rate measured using a known method. The rates were varied to 7.3%, 8.0%, 12.0%, 13.6%, 15.3% and 18.0% respectively, at intervals of about 15 to 20 seconds. Each step is clearly visible after only a few seconds during which the applicator is settling and the averaging step is taking place.

Figure 8 shows a plot of the moisture content of a building board against time, as the board exits a dryer. It can be seen in Figure 8 that the plot is generally constant but has regular spikes of particularly high moisture content. These relate to areas of the board exiting a dryer whose local composition has affected the drying rate. It is clear from figure 8 that individual boards whose drying has been unsatisfactory can be identified and subjected to remedial action, if necessary.

Figure 9 shows similar data that of figure 8, but for an alternative form of board referred to as "flake board". Again, individual boards with too much moisture can be clearly distinguished.

Figure 10 shows a portion of figure 9 on a much larger time scale. At this scale, individual boards can be resolved and the moisture profile along the board identified.

Figure 11 shows the output of a sensor installed so as to follow the hydration reaction of a gypsum sample. It is clear from Figure 11 that the initial sample is steady at a rate of about 0.020, but after 30 minutes or so the attenuation rate is a steady 0.090. It is therefore possible to monitor the attenuation rate and deduce that the reaction has completed once the attenuation rate is steady.

There will usually be a phase shift between the transmitted and received signals. This could cause difficulties in measuring absolute values
of the signal strength. It is therefore usually simpler to assume a fixed and stable strength for the transmitted signal and measure only the received signal. This gives acceptable results in practice. It may be necessary to recalibrate the assumed value on an occasional basis.

The phase shift can be used to infer the position and geometry of the substrate by comparing the phase shifts of the two transmitted signals. This information can be used to compensate the final output reading.

It will of course be appreciated by those skilled in the art that the above-described embodiments are given by way of illustration of the invention, and that many variations thereto are possible whilst remaining within the scope of the invention.
CLAIMS

1. A method of determining the quantity of unbound water in a substrate, comprising directing a beam of incident microwave radiation of a wavelength absorbable by the unbound water; measuring the microwave radiation transmitted through the substrate, and determining the quantity of water present from the intensity difference between the incident radiation and the transmitted radiation.

2. A method according to claim 1 wherein the frequency of incident microwave energy used is 3 GHz or greater.

3. A method according to claim 1 wherein the frequency of incident radiation lies in the range of 3 to 30 GHz.

4. A method according to claim 1 wherein the frequency of incident radiation lies in the range 9 to 13 GHz.

5. A method according to claim 1 wherein the frequency of incident radiation lies within the range 10 to 12 GHz.

6. A method according to claim 1 wherein the frequency of incident radiation lies within the range 10 to 11 GHz.

7. A method according to any preceding claim wherein the incident power is less than 20 mW.

8. A method according to any one of claims 1 to 6 wherein the incident power is less than 15 mW.

9. A method according to any one of claims 1 to 6 wherein the incident
power is about 8mW.

10. A method according to any one of claims 1 to 6 wherein the incident power is less than 0.6 Wcm$^2$.

11. A method of determining the quantity of unbound water in a substrate, comprising directing a carrier wave of incident microwave radiation of a wavelength absorbable by the unbound water, together with a superimposed low frequency reference signal; measuring the strengths of the carrier wave and reference signal after transmission through the substrate, and determining the quantity of water present from the intensity difference between the carrier wave and the reference signal.

12. A method according to claim 11 wherein the frequency of the reference signal is not greater than 100 kHz.

13. A method according to claim 11 wherein the frequency of the reference signal is between 10 Hz and 10 KHz.

14. A method according to claim 11 wherein the frequency of the reference signal is about 1kHz.

15. A method according to claim 11 wherein the frequency of the reference signal is about 81 Hz.

16. Apparatus for determining the level of unbound water in a substrate, comprising a microwave transmitter; a microwave receiver; means for comparing the relative intensities of the incident radiation and transmitted radiation, and converting the difference into a displayed value indicative of water content; and a display for displaying the said value.
17. Apparatus according to claim 16 wherein the microwave transmitter is adapted to emit a carrier signal and a superimposed reference signal, the reference signal being of a frequency not substantially absorbable by either water or the substrate, the means for comparing the relative intensities being adapted to compensate for signal dissipation through use of the reference signal strength.

18. A method of determining the level of unbound water in a substrate substantially as herein described with reference to and/or as illustrated in the accompanying drawings.

19. Apparatus for determining the level of unbound water in a substrate substantially as herein described with reference to and/or as illustrated in the accompanying drawings.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6    GOIN22/04

According to international Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6    GOIN

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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| Patent family members are listed in annex |

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Date of the actual completion of the international search

9 October 1997

Date of mailing of the international search report

30/10/1997

Name and mailing address of the ISA

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