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Magnetic Resonance Techniques and Food Engineering

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Abstract

Magnetic resonance techniques including nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI) are used as new and powerful sensor technology. NMR is used for acquiring time and frequency domain information nondestructively from biosystem. MRI is intensively used in medical area and has been developing as a technique for studying the unit operations during food processing. Phenomena such as phase changes, transport properties, and interactions which determine the structure and stability of particular foods can be analyzed. These techniques are presently utilized as a tool to refine theoretical models for improving food processes such as canning, freezing, frying, drying, form and emulsion formulation, crystallization, and extrusion processes. Also, they are being used as a nondestructive tool for detecting internal quality factors of food and agricultural products, as well as quality factors such as bruises, dry regions, worm damage, stage of maturity, and presence of voids, seeds, and pits of food and agricultural products. These techniques are now being utilized in developing on-line quality monitoring and evaluating sensors.

Key words: nuclear magnetic resonance (NMR), magnetic resonance imaging (MRI), food, nondestructive, sensor

Introduction to Nuclear Magnetic Resonance (NMR) and Magnetic Resonance Imaging (MRI)

The significant advantage of Nuclear Magnetic Resonance (NMR) spectroscopy is that it is a nondestructive method. A variety of NMR techniques exist which can provide structural information. High resolution NMR spectroscopy is a basic tool for biologists and chemists. This technique is often used to measure distances between atoms. Magnetic resonance imaging (MRI), an extension of 2-D NMR spectroscopy was initially developed as a medical diagnostic tool. MRI is successful in the medical field and has been recently applied in the biological and agricultural area due to the advance of computer technology and the potential for low-cost MRI system manufacture. MRI provides the macroscopic spatial distribution of information based on the chemical and electronic environment of nuclei within a sample.

Chen *et al.* (1989) demonstrated that MRI can be used for evaluation of various internal quality factors of fresh fruits and vegetables. NMR is not harmful to products and does not damage product quality, and can be used in examining food stability and structure, moisture migration, rheology, phase changes, *etc.* (McCarthy, 1994; Kauten and McCarthy, 1995; Hills, 1995; McCarthy and McCarthy, 1996). Rapid MRI techniques should provide a basis for on-line product sorting based on internal quality of the product (Chen *et al.*, 1996; Kim *et al.*, 1999)

There are several NMR sensitive nuclei such as ^{1}H, ^{13}C, ^{19}F, ^{23}Na, ^{31}P, and ^{39}K that are found in most of biological materials. Among these nuclei ^{1}H is the most abundant in all biological materials (Gadian, 1995). In this study, therefore, the discussion about NMR and MRI will be focused on ^{1}H.

A. NMR Spectroscopy

1) Nuclear Magnetic Resonance (NMR) Principles

When nuclei with a magnetic moment, like protons, are placed in an external magnetic field the sample

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develops a net macroscopic magnetization, **M**. This magnetization is generated along the direction of the applied field. The magnetic moments of these nuclei tend to align along the magnetic field direction and precess about their own axes at a specific frequency (ω_0) which is related to the magnitude of applied external magnetic field. The frequency can be expressed as the following equation known as the Larmor relationship:

$$\omega_o = \gamma B_o(\text{rad/s}) \Rightarrow \nu_o = \frac{\gamma}{2\pi} B_o(\text{Hz}) \quad (1)$$

where γ in$\text{rad} \cdot \text{s}^{-1} \cdot \text{Gauss}^{-1}$ and B_0 in Gauss. The magnitude of **M** is proportional to the number of proton (^{1}H) in a sample. The gyromagnetic ratio is constant and varies with a nucleus (γ of ^{1}H is $2.68\times10^4 \text{rad} \cdot \text{s}^{-1} \cdot \text{Gauss}^{-1}$). **M** is detected with the aid of an additional radio frequency (RF) pulse ($\mathbf{B}_1$) excitation. The role of $\mathbf{B}_1$ is to nutate **M** to *xy* plane where NMR signal can be acquired. Following an external excitation, the signal induced in the receiver coil decays exponentially with respect to time. This phenomenon is called relaxation. During relaxation the spins to return to their equilibrium energy levels by transferring energy into the surroundings. Relaxation is described by two processes. One is longitudinal relaxation (spin-lattice or T_1 relaxation) and the other is transverse relaxation (spin-spin or T_2 relaxation).

From equation (1) identical nuclei precess at the same frequency in the same externally applied magnetic field. In a real sample the local magnetic field at each nucleus varies as a result of the influence of the local chemical and electronic environment on the applied magnetic field. Therefore, the total effective magnetic field, B_{eff}, is

$$B_{eff} = B_o(1-\sigma) \quad (2)$$

where σ is shielding constant. Combining equation (1) and equation (2) results in a new expression:

$$\nu_o = \frac{\gamma}{2\pi} B_o(1-\sigma) \quad (3)$$

The magnitude of σ is related to the chemical environment of the nucleus, therefore, proton (^{1}H) nuclei in oil, sugar, and water generate signals at different frequencies. The chemical shift is defined as the resonance frequency separation from an arbitrarily chosen reference frequency and is expressed in terms of ppm (1 ppm= $\frac{\nu - \nu_{ref}}{\nu_{ref}} \times 10^6$) of the applied magnetic field (Gadian, 1995). An example of chemical shift in a ^{1}H NMR spectrum is shown in Figure 1. From Figure 1, the resonance frequency separation of oil and water from an avocado spectrum is 3.5 ppm and sugar and water from a plum spectrum is 1.1 ppm.

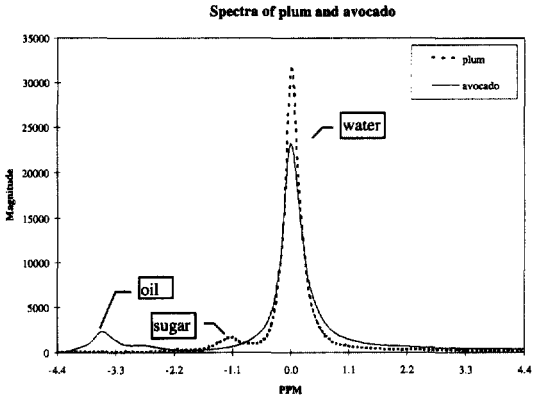

Fig. 1. Superimposed spectra of an avocado and a plum showing oil and water resonance peaks from an avocado and sugar and water resonance peaks from a plum.

2) Spectroscopy

After $\mathbf{B}_1$ excitation a signal, s(t), is induced in the receiver coil. The signal, s(t), decays at a rate of T_2^* and expressed in the following equation:

$$S(t) \propto \sum_i \rho_i \exp(-j\omega_i t)\exp(-t/T_{2_i}^*) \quad (4)$$

where i is the index of a component, and j indicates imaginary number. This signal is known as the Free Induction Decay (FID).

A NMR spectrum signal, S(ω),obtained by a Fourier transform of an FID signal, s(t), is a plot of the amount of energy absorbed at a specific frequency, and expressed as:

$$s(\omega) \propto \sum_i \rho_i \left[\frac{T_{2_i}^*}{1 + (\omega_i - \omega)^2 T_{2_i}^{*2}} \right] \quad (5)$$

The intensity of $S(\omega)$ is influenced by $T_{2_i}^*$, ρ_i, and chemical shift effect of a component i (Gadian, 1995). Figure 1 displays typical spectra of an avocado and a plum. In the avocado spectrum water and oil compo-

nents have separate resonance peaks. In the plum spectrum water and sugar components have separate resonance peaks but they very close to each other. Therefore, higher magnetic field is required to improve the resolution of different components based on their chemical shifts.

B. Magnetic Resonance Imaging (MRI)

1) MRI Principles

MRI is a collection of experimental techniques, which are designed to allow one to measure the NMR properties of a sample as a function of spatial position. The experiment proceeds by placing the sample in a homogeneous external magnetic field. Additional pulsed linear magnetic field gradients are used to produce a frequency variations across the sample which can be converted into spatial coordinates. The relationship between frequency and magnetic field is:

$$\omega = \gamma(B + Gx) \tag{6}$$

where ω is the Larmor frequency (rad/sec), G is the linear magnetic field gradient (in Gauss/cm) and x is the spatial distance (cm). For instance, if a linear gradient G is applied in the x direction then the precessional frequency becomes a function of the sum of the homogeneous field and the linear gradient. The frequency spectrum can easily be converted into a position-dependent signal intensity. By the proper application of gradient one-, two- or three-dimensional mappings of the NMR signal intensity can be recorded (McCarthy, 1994).

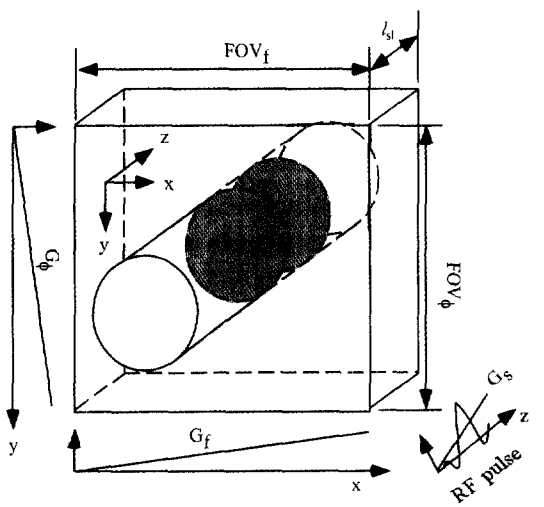

Fig. 2. Diagram showing slice selection, phase encoding, and frequency encoding gradients, and field of views in frequency and phase encode directions, FOV_f and FOV_ϕ. Gray volume indicates slice selected region where thickness is l_{sl} in a cylindrical sample.

2) NMR Image Generation

There are three basic terms to understand MRI: slice selection, phase encoding, and frequency encoding. For this paper slice selection will be associated with the z- direction, phase encoding with the y-direction, and frequency encoding with the x-direction as shown in Figure 2.

To localize NMR signals along the z-direction in a sample, slice selection is performed. To select an orthogonal plane to the z-direction with a certain slice thickness, l_{sl}, a shaped RF pulse and a linear gradient along the z-direction are used as shown in Figure 2. The NMR signal is encoded with the y-position by using phase encoding. The phase of NMR signal is a function of y and expressed as follows:

$$\phi(y) = \gamma G_\phi t_\phi y \tag{7}$$

where G_ϕ is magnitude of gradient along phase encode direction, and t_ϕ is phase encoding time. The NMR signal along the x-direction is encoded in terms of frequency. The frequency dependence of the NMR signal is expressed as:

$$\omega(x) = \gamma G_f x \tag{8}$$

where G_f is magnitude of gradient along frequency encode direction.

To generate a two-dimensional static magnetic resonance image, the time domain data S(t) is arranged as a frequency dependent data set, $S(k_x, k_y)$ in k-space,

$$S(k_x, k_y)=$$

$$\iiint \rho(x, y, z)dz \exp[i2\pi(k_x x + k_y y)]dxdy \tag{9}$$

where $k_x=(2\pi)^{-1}\gamma G_f t_f$, $k_y=(2\pi)^{-1}\gamma G_\phi t_\phi$, and t_f is dwell time (Callaghan, 1991). The data set $S(k_x, k_y)$ is Fourier transformed to obtain a proton density two-dimensional image,

$$\rho(k, y)=$$

$$\iint S(k_x, k_y)\exp[-i2\pi(k_x x + k_y y)]dk_x dk_y \quad (10)$$

Field of views of a magnetic resonance image in frequency encode direction (FOV_f) and phase encode direction (FOV_ϕ) can be controlled in the following manner:

$$FOV_f = (2\pi)(\gamma G_f t_f)^{-1} \quad (11)$$

and

$$FOV_\phi = (2\pi)(\gamma \Delta G_\phi t_\phi)^{-1} \quad (12)$$

where ΔG_Φ is gradient increment along phase encode direction.

3) Contrast in NMR Images

In MRI, a spin-echo is very important MR phenomenon used to acquire an MR image. The amplitude of the MR signal which is proportional to the concentration of mobile protons at a certain point in a sample causes the intensity or brightness at that position in a magnetic resonance image. Mobile proton density, and relaxation times T_1 and T_2 which are sample-dependent parameters are good sources for generating contrast in an MR image. There are two important experimental parameters used to acquire an MR image. These are repetition time (TR) which is the time interval from the beginning of a pulse sequence until the beginning of the next pulse sequence, and echo time (TE) which is time gap from the first radio frequency (RF) pulse of a pulse sequence to the middle of a spin echo (Callaghan, 1991). These parameters can be varied to provide contrast that describes the structure of a sample. Table 1 describes three types of spin echo images, proton density, T_2 weighted, and T_1 weighted images, generated by changing TR and TE. The spin echo image signal, S(t), is a function of proton density, T_1 relaxation and T_2 relaxation:

$$S(t) \propto \sum_i \rho_i \exp(-TE/T_{2i})(1 - \exp(-TE/T_{1i})) \quad (13)$$

where *TE* is echo time, *TR* is repetition time and *i* is a component index.

Table 1. Contrast sources for spin echo NMR images

Image	TE	TR	Contrast
Proton density	$<< T_2$	$>> T_1$	Proton density
T_2 weighted	$\sim T_2$	$>> T_1$	T_2
T_1 weighted	$<< T_2$	$\sim T_1$	T_1

If both TR and TE have values comparable to T_1 and T_2 respectively it is called mixed T_1 and T_2 weighted imaging. However short TRs used together with long TEs lead to an overall decrease in signal intensity, which is not usually desirable. Generally, TR is used to control T_1 weighting and TE is used to control T_2 weighting. Short T_2 tissues are dark on T_2 weighted images, but short T_1 tissues are bright on T_1 weighted images. T_1 and T_2 weighting is applied only to samples with multiple values of T_1 and T_2, hence the objective is to enhance the signal from one component relative to the other.

Additional factors affecting the contrast of an MRI image are chemical exchange of molecules, inhomogeneity of local magnetic field due to variations in the diamagnetic susceptibility within the sample (for example, intercellular liquids and intracellular air pockets have different susceptibility), and motion of spins through spatial variations in the magnetic field.

Applications of NMR Techniques in Food Reasearch

Several recent advances in NMR and MRI published after the review by Schmidt and Lai (1991) and McCarthy (1994) will be highlighted. These recent advances are in sensor development and structure determination.

The interest in using tomographic sensors to control industrial processes has interested in the last 10 years. Unique aspects of MR tomography include sensitivity to a variety of physical and chemical properties, measurements are rapid and the signal is based primarily on the internal properties of the sample. Two important issues in the development of MR based sensors are the influence of motion and construction of a low-cost flexible system. In addition to measuring physical properties MR can be used to quantify structure. A quantitative measure of food microstructure is important for understanding macroscopic properties like viscosity, permeability and texture. Recent advances in the use of NMR techniques to monitor food processing include viscosity measurement of fluid and examination of mixing process.

1. Measurement of Quality

1.1 Maturity of fruits

As fruits mature they have more water and oil, which can be detected by the MR techniques. Figure 3 shows two kinds of kiwi fruits, one is immature and the other is mature. There are three clear layers regardless of their degree of maturity. However, the center part (refer arrows) of an immature kiwi fruit generates less signals than that of a mature one. This indicates that MRI can be used for determining kiwi maturity.

1.2. Cherry Structure

Using a inversion recovery imaging pulse sequence, 1-D images of a cherry with a pit at various inversion recovery (IR) times, 100 ms, 200 ms, 300 ms, 400 ms, 500 ms, 600 ms, 664 ms, 700 ms, 800 ms, 900 ms, 1000 ms, 1200 ms, 1500 ms, 2000 ms, 3000 ms, and 5000 ms were acquired. Figure 4 shows that the cherry flesh signal can be suppressed with a suitable recovery time and hence the presence of a pit can be directly detected. In this case an IR time of 664 ms is suitable for pit detection.

Applications of imaging methods contrast to spectral methods in that acquisition times are short, typically on the order of 10 ms. It is the time delays in the pulse sequence which limit the throughput. Two approaches can be taken to increase throughput, either image multiple fruits or separate radio frequency/gradient coil assemblies for different parts of the sequence. Separate radio frequency/gradient coil assemblies are obviously not the preferred approach since the capital costs and engineering/design costs would rise rapidly.

2. Determination of Sample Structure

2.1 Cheese

In the producing and marketing of cheeses, cheese quality evaluation is very important. The quality of cheese is determined by its structure and body features.

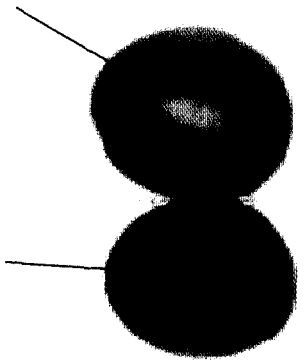

Fig. 3. Image of immature (top) and mature (bottom) kiwis. MRI parameters: TR=250 ms and TE=50 ms

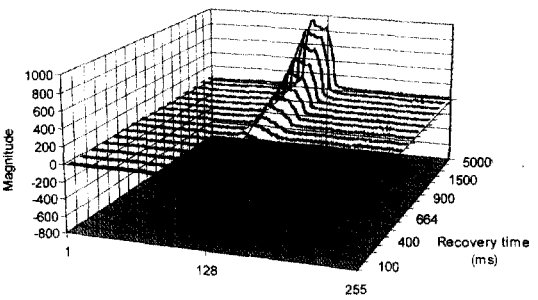

Fig. 4. Real parts of 1-D images of a cherry with various recovery times generated by the inversion recovery pulse sequence.

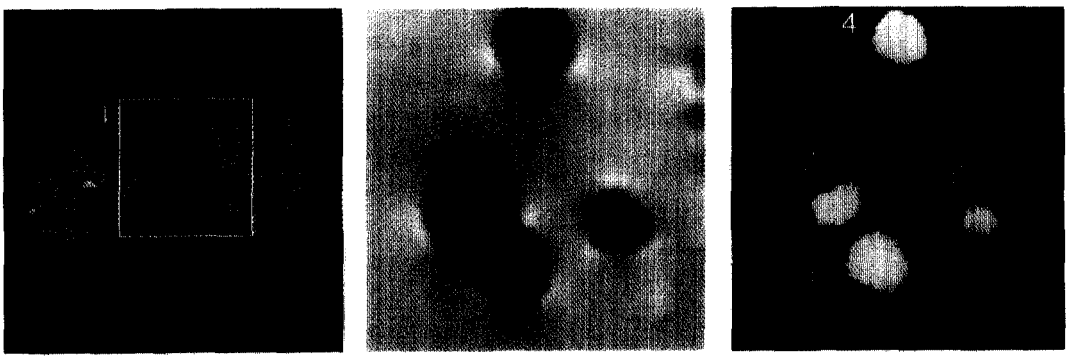

Fig. 5. Magnetic resonance image of Swiss cheese showing 100×100 pixel subimage (left), zoomed subimage for texture analysis (center) and processed binary subimage for structure analysis (right).

evaluating cheese internal structure is essential.

Rosenberg *et al.* (1991) reported that MRI showed a high potential as a nondestructive method to evaluate the internal characteristics of Swiss-type cheese. MRI can be used for detecting defects such as small eyed, large eyed, and, frog mouth, which are critically important for grading cheese.

The structure of eyes and the internal texture of Swiss-type cheeses were nondestructively measured on from magnetic resonance images. Figure 5 shows a procedure for processing of a two-dimensional magnetic resonance image of Swiss cheese to obtain internal structure and texture. The first step is to extract a 100x100 subimage from a two-dimensional image. The second step is to calculate mean gray value, standard deviation, angular second moment and contrast of gray scale image to define the internal texture of Swiss cheese. The third step is to extract a binary image of eyes from a gray scale image by a thresholding method. The last step is to determine the number of eyes, thinness ratio, cross-sectional area, and the eye center location by chain coding and labeling methods for identifying eye structure. Magnetic resonance imaging combined with image and texture analysis has a high potential for automatic nondestructive quality evaluation of Swiss-type cheeses based on classification of eyes and internal structure. This grading protocol will serve as a model for other types of processed foods (Kim *et al.*, 1996).

2.2. Gels

Characterization of the microstructure of hydrogels by SEM and similar techniques can usually only be accomplished after a preparation procedure which may alter the structure of the biopolymer networks. NMR relaxation techniques have been applied to study gels and it has been shown that relaxation processes are sensitive to the morphology of gels (Belton, Hills, and Raimbaud 1988). Traditional pore geometries used to interpret the relaxation measurements are spherical, cylindrical or planar. These geometries are not suitable to characterize the fibrous network in low solids fraction hydrogels. A more appropriate geometrical model is one based on cylindrical single fibers, a fiber-cell model (Chui, Phillips, and McCarthy 1995). The relaxation times and the model are used with the magnetization-diffusion equation to determine pore radius distributions. This method will be useful to measure the microstructure and changes in the microstructure occurring over length scales from nm to 50 μm. This technique is able to monitor changes in microstructure induced by processing operations like freezing.

3. Single Cell Imaging

Interpretation of NMR data from groups of intact cells is complicated because an average measurement is obtained (Aiken, Hsu and Blackband, 1995). Improved interpretation can be obtained if the NMR characteristics of the individual components in cell assemblies were known. This is the basic motivation for NMR imaging of single cells. The NMR characteristics of clustered cells are:

- mono exponential spin-lattice relaxation
- multi exponential spin-spin relaxation
- lower intracellular diffusivities than extracellular diffusivities.

The mechanisms responsible for this relaxation and diffusion behavior is not completely understood. Additionally, how relaxation and diffusion behavior change as a function of physiological and/or processing conditions is only just beginning to be examined (Aiken, Hsu, and Blackband 1995). A detailed review of single cell NMR imaging studies has recently been completed by Aiken and coworkers (Aiken, Hsu, and Blackband 1995). Studies to date have included both plant and animal cells. Of importance to food scientist is the ability to directly measure cell wall permeability and intracellular restructuring under simulated storage and processing conditions. Additional details can be found in the review by Aiken, Hsu, and Blackband (1995).

4. Food processing monitoring

NMR techniques can quantify various physiochemical quantities such as moisture content, temperature, viscosity, and food quality related to food processing and storing. Studies of velocity profile of fluid, temperature mapping of drying and freezing operation, and mixing of materials with aids of NMR techniques give valuable information to understand phenomena happened in food research. Figure 6(a) shows velocity profile of water

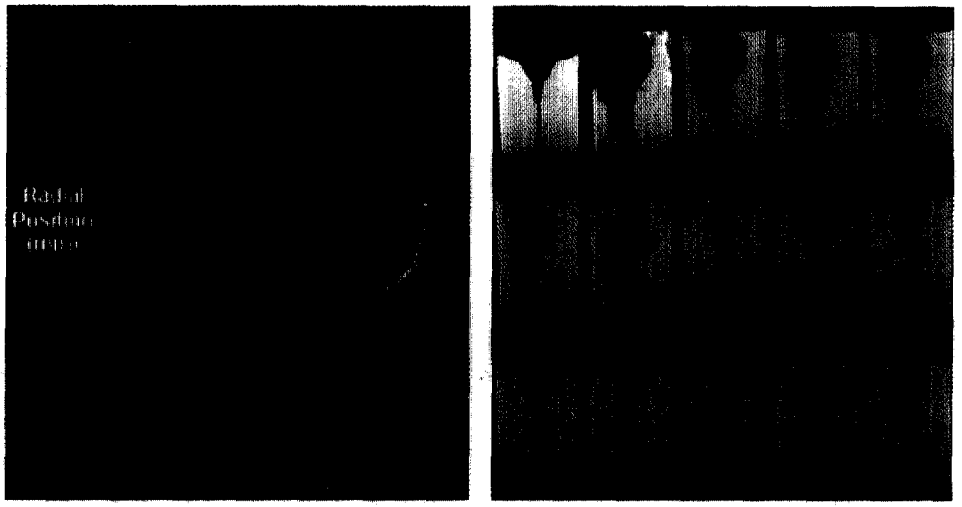

Fig. 6. Magnetic resonance images of velocity profile of water (a) and mixing of particle and fluid (b) (courtesy of Prof. M.J. McCarthy).

flowing through a pipe acquired using an MRI flow imaging technique. With simultaneous measurement of pressure drop of the flow, shear viscosity of flowing material could be obtained without disturbing flow system. Figure 6(b) shows series of magnetic resonance images of mixing of particles and fluid in a stirred vessel. This characterizes the mixing of particles and fluid under laminar mixing condition and observes structures that develop during the mixing process.

5. NMR sensor

A high speed NMR quality evaluation sensor was designed, constructed and tested (Chen *et al.*, 1996; Kim *et al.*, 1999. The feasibility test of NMR sensor was performed with durian fruits. Spectra of each fruit were acquired at three different speeds, 100, 200, and 300 mm/s. From durian spectra three magnetic resonance peaks, water (at 0 ppm), sugar (at −1 ppm), and oil (−4 ppm), were observed. With a higher conveyor moving speed the magnitude of magnetic resonance peak of water component was decreased. However, still there are clear three resonance peaks. If these three resonance peaks are related to the fruit maturity they can be used as quality factors for on-line NMR sensors (refer Figure 7).

One needs to recognize that the requirements for spectra for process control are different than for classical

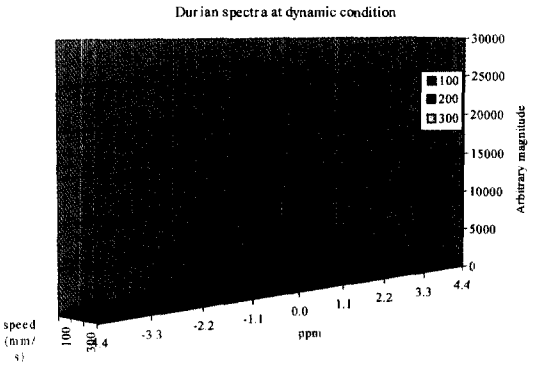

Fig. 7. Durian spectra acquired at three different speeds, 100, 200, 300 mm/s.

analytical spectroscopy. By the nature of the application the material to be examined will be moving. Motion will result in apparent changes in the relaxation rates (Tellier and Mariette, 1995). The changes will effectively tend to reduce spectral resolution and the signal-to-noise ratio.

The reduction has not significantly influenced the quantization of the durian data. Details on conditions for quantitative analysis of flow can be found in the chapter by Tellier and Mariette (1995). The reduction in spectral resolution resulting from the motion of the object is actually advantageous for process control. A faster relaxation rates results in faster data acquisition and hence increased throughput rates. Spectral data should

be acquired for at least 4 T_2* to ensure the entire decay is recorded. Thus, any increase in decay rate results in a four folds decrease in acquisition time. This also has an influence on the design of the main magnetic field in terms of homogeneity to optimize throughput.

Conclusions

Magnetic resonance techniques including NMR and MRI are powerful tools for the study of food systems due to their nondestructive and noncontacting nature. The physical and chemical properties of foods and characterization of food processing could be obtained without disturbing the system. NMR and MRI sensors could be used as on-line process control sensors to measure internal properties quantitatively. Internal structure information of foods obtained by NMR and MRI provides a better understanding of food process. Microscopic imaging capability of NMR enables food researchers to directly measure the structure of cell wall. MRI information combined with classical analysis techniques provides a more complete understanding of structure and chemical composition of food systems.

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