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Prototype of Non-Destructive Fruit Sweetness Sensor

Lecture No 9211-18

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SUMMARY:

A model LL-4 magnetic resonance low level unit designed to operate at 5.35 MHz was used to perform proton magnetic resonance (1H-MR) tests on sugar solutions. The magnet console was designed and built by Agricultural Engineering Department personnel. The homogeneity of the magnetic field is critical to achievement of a good signal and to good performance of the device. Measurements of magnetic field strength along the major axes of the air gap are reported. Test tubes were filled with sugar solutions and placed in the air gap between the permanent magnets in the magnet console. Sugar solutions containing 0% to 30% sucrose were interrogated using the Hahn spin echo pulse sequence. The amplitude of the echo peak was well correlated with percent sugar content in solutions.

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INTRODUCTION

Sweetness is one of the more important quality attributes of fruits and vegetables. Noninvasive sorting of fruits according to sugar content would enable retailers and wholesalers a wider range of marketing options. For fruits in which sugar content changes with ripenes, the ripest fruits could be sorted out for immediate utilization or diversion to processing. Grocery stores could display fruits according to sugar content. This would allow consumers to purchase fruits which suit their individual preferences for sweetness or which are appropriate for the use intended. High sugar content fruits could be sold for a premium.

Proton magnetic resonance (¹H-MR) has been used to measure the sugar content of solutions (Mora-Gutierrez and Baianu, 1989), fruit tissue (Cho et al., 1991), and in-tact fruits (Stroshine et al., 1991). This paper describes experiments which focused primarily on measurements with sugar solutions using the Hahn spin-echo pulse sequence. This sequence has not been used in previous tests (Cho et al., 1991; Stroshine, et al., 1991). The experiments were performed in a low resolution device which used a permanent magnet and operated at a frequency of 5.35 MHz. The objective of the research was to evaluate the ability of the pulse technique and the device to distinguish the sugar content of solutions containing between 0 and 35% by weight sugar. This is the range of sugar content found in fresh fruits and vegetables.

MATERIALS AND METHODS

A schematic of the low resolution ¹H-Magnetic Resonance equipment is shown in Figure 1. The circuitry for the device is similar to that described by Clark (1964) and Clark and McNeil (1973). The electronic components were built by the Southwest Research Institute (SWRI) of San Antonio, Texas. Components were adapted to operate with a magnet console that had been previously designed by project personnel. SWRI also supplied the software which controlled the electronic components, digitized the magnetic resonance signal, and determined various signal parameters (e.g. peak height).

Console design is described by Cho, et al. (1990). It has an air gap of 10.16 cm. A coil was wrapped around a glass tube having an inside diameter of 3.80 cm. The tube and coil were then mounted in a shielding box and inserted into the air gap. The magnetic field strength in the center of the air gap is 1256 G and the frequency of the RF signal is therefore approximately 5.35 MHz.

Homogeneity of the magnetic field within the console is essential to optimum performance of magnetic resonance instruments. Therefore, the magnetic flux density at various points within the console was measured using an RFL Model 912 Gaussmeter and a Model 912-039 Hall effect probe (Magnetic Instrumentation, Inc., Indianapolis, Indiana). The probe was calibrated so that it gave maximum sensitivity at 1256 gauss, the desired field strength for the console. The probe calibration was periodically checked using a zero gauss chamber and a reference magnet.

Measurements were taken as follows. The probe was positioned so that it was in the center of the air gap and halfway between the ends of the coil. It was oriented in such a manner that it measured flux density perpendicular to the magnet faces. The centered position was taken as the origin of an x-y-z coordinate system. The flux density at this origin was measured and used as the baseline from which relative field changes were determined. The y-axis of the coordinate system was parallel to the axis of the RF coil. The z-axis was perpendicular to the face of the magnets, i.e. in the direction of B_0 , the permanent magnetic field which acts upon the sample when it is inserted in the console. The flux density at points along the y axis was determined by first centering the Hall effect probe in the glass sample tube and then moving it along the tube axis. For measurements at points along the x and z directions, the shielding box containing the sample tube and coil were removed. The Hall effect probe was held in place using a ring stand and clamp. For the x-axis measurements, the base of the ringstand was moved the appropriate distance and for the z-axis measurements, the clamp was moved up or down on the ringstand.

Console performance was evaluated using glass culture tubes containing sucrose solutions varying in sugar content (percent by weight) from 0 to 35%. The culture tubes were 16 mm in diameter and 150 mm long and had plastic screw-on caps. The sugar solutions were mixed and then filtered through a nylon membrane filter (0.2 µm pores). The tubes were autoclaved before filling to reduce the likelihood of microbial activity within the tubes. During the experiments, the tubes were inserted into the console parallel to the y-axis. (See the description above regarding magnetic flux density measurements.) The tubes were completely filled so as to eliminate air. The tubes either contained no air bubble, or a very small air bubble. This was perceived as important because it is believed that air bubbles can affect magnetic resonance signals.

A second console evaluation used hollow plastic spheres 25mm in diameter (Total Plastics, Inc., Fort Wayne, Indiana). The spheres were filled with filtered sugar solutions containing 0, 1, 2, 3, 4, 5, 10, and 15% by weight sucrose. These spheres are approximately the same diameter as a sweet cherry. The spheres were positioned in the center of the glass sample tube using a cylindrical teflon sample holder. A 25mm diameter hole was drilled perpendicular to the axis of the cylinder and the sphere was placed in the hole.

The sample was interrogated using the Hahn Spin-echo pulse sequence, 90° - τ -180°, (Hahn, 1950). The 90° pulse was of duration 6 µsec and the value of τ , the delay time between the 90° and 180° pulses, was 100 msec. The Hahn echo sequence produces an echo peak at time 2τ . For a given sample, the height of the echo peak is affected by τ . Preliminary tests showed that a τ of 100 msec gave the best differentiation among the various sugar solutions.

Just prior to the tests, the equipment was tuned by inserting a water sample and viewing the FID signal on an oscilloscope. The magnetic field strength was adjusted by varying the current supplied to shimming coils wrapped around the poles of the permanent magnet. The magnetic field strength was adjusted so that the FID signal was a smooth decay curve. After a sample was inserted, the pulse sequence was applied and the system software determined the amplitude of the echo peak.

RESULTS

Figure 2 shows the deviations of magnetic field from 1256 gauss at various points along the x y and z axes (as defined in Materials and Methods) of the magnet console. The inhomogeneity was less than 300 ppm. Within several mm of the center, the magnetic field homogeneity in the x and z directions was quite good. The greatest variation in the magnet field was in the y direction. The long axis of the culture tubes coincided with the y-axis while the diameter of the tubes was relatively small. Therefore, the yaxis variations may have had a significant effect on these experiments. The effect should have been less significant for the subsequent experiments with the plastic balls.

Figure 3 shows the relationship between the echo peak amplitude and the sugar content of the solutions in the culture tubes. Three consecutive interrogations were performed on each sample and the result of each interrogation was recorded separately. In general, there was a good agreement among replicate interrogations of the same sample. With the exception of the tests on the 10% sugar solution, there appears to be a relatively linear relationship between peak height and sugar content. The r^2 for a linear regression of the data in Figure 3 was 0.86.

For the tests on the 25 mm diameter plastic spheres, the relationship between echo peak amplitude and percent sugar in the solutions is shown in Figure 4. The sugar content of these seven samples was 15% or less. Five of the samples had sugar contents less than 5%. Even at these relatively low sugar contents, there appeared to be a reasonable correlation between echo peak height and sugar content. The r^2 for a linear regression of the data in Figure 4 was 0.82.

One of the uncertainties in the experiment was an observed change in the magnet temperature. This was caused by the heating effect of the Stroshine, et al.

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shimming coil. This heating reduced the magnetic field homogeneity which in turn affected the signal. Another uncertainty was the precision in tuning of the system. Equipment and procedural modifications are currently being developed which would minimize these problems. For example, redesign of the shimming coils could reduce the amount of resistance heating. Magnet temperature could be stabilized using a heating element coupled to a temperature controller. We anticipate that these and other changes will further improve the correlations between sugar content and peak height. We have also conducted preliminary tests with brine sweet cherries, cherry tomatoes, and core sections of apples and pears. Correlations between refractive index and echo peak height appear to be similar to those achieved with sugar solutions.

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CONCLUSIONS

The magnetic console design proposed by Cho, et al. (1990) provides acceptable homogeneity. It performed satisfactorily when used with a low field magnetic resonance device. Additional modifications to the console and changes in experimental procedures should improve performance. Experiments with the device indicated that the Hahn spin echo pulse sequence can be used to determine the sugar content of sugar solutions. It appears possible to extend the technique to measurement of sugar content in fruits and vegetables.

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REFERENCES

Cho, S.I. 1989 Development of a Nuclear Magnetic Resonance Based Sensor to Detect Ripeness of Fruit. Unpublished PhD dissertation, Agricultural Engineering Department, Purdue University, West Lafayette, IN.

Cho, S.I., V. Bellon, T. M. Eads, R.L. Stroshine, and G.W. Krutz. 1991. Sugar content measurement in fruit tissue using water peak suppression in high resolution ¹H Magnetic Resonance. *Journal of Food Science* 56(4):1091-1094.

Cho, S.I., G.W. Krutz, H.G. Gibson, and K. Haghighi. 1990. Magnet Console Design of an NMR-Based Sensor to Detect Ripeness of Fruit. *Transactions of ASAE* 33(4):1043-1050.

Clark, W.G. 1964. Pulsed Nuclear Resonance Apparatus. *The Review of Scientific Instruments* 35(3):316-333.

Clark, W.G. and J.A. McNeil. 1973. Single Coil Series Resonant Circuit for Pulsed Nuclear Resonance. 1973. *The Review of Scientific Instruments* 44(7):844-851.

Hahn, E.L. 1950. Spin Echoes. *Physical Review* 80(4):580 to 594.

Martin, M.L., J.J. Delpoech and G.J. Martin. 1980. **Practical NMR Spectroscopy.** Heyden and Son, Ltd., London.

Mora-Gutierrez, A., and I.C. Baianu. 1989. ¹H NMR Relaxation and Viscosity Measurements on Solutions and Suspensions of Carbohydrates and Starch from Corn. *Journal of Agricultural Food Chemistry* 37(6):1459-1465.

Stroshine, R.L, S.I. Cho, W.K. Wai, G.W Krutz, and I.C. Baianu. 1991. Magnetic Resonance Sensing of Fruit Firmness and Ripeness. Paper 91-6565. American Society of Agricultural Engineers, St. Joseph, Michigan 49088-9659



Figure 1. NMR Fruit Ripeness Sensor System Block Diagram.

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Figure 2. Deviations of the magnetic field from 1257 gauss along the x, y, and z axes of the magnet console.

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Figure 3. Relationship between height of the Hahn Spin echo peak (volts) and the sugar content (% by weight) for sugar solutions varying in sugar content from 0 to 35%.



Sugar Content (% by weight)

Figure 4. Relationship between normalized height of the Hahn Spin echo peak (millivolts per gm of solution) and the sugar content (% by weight) for plastic balls containing sugar solutions varying in sugar content from 0 to 15%.

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