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Battery-free radio frequency identification (RFID) sensors for food quality and safety

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Abstract

The market demands for new sensors for food quality and safety stimulate the development of new sensing technologies that can provide an unobtrusive sensor form factor, battery-free operation, and minimal sensor cost. Intelligent labeling of food products to indicate and report their freshness and other conditions is one of important possible applications of such new sensors. We have applied passive (battery-free) radio frequency identification (RFID) sensors for highly sensitive and selective detection of food freshness and bacterial growth. In these sensors, the electric field generated in the RFID sensor antenna extends out from the plane of the RFID sensor and is affected by the ambient environment providing the opportunity for sensing. This environment may be in the form of a food sample within the electric field of the sensing region or a sensing film deposited onto the sensor antenna. Examples of applications include monitoring of freshness of milk, freshness of fish, and bacterial growth in a solution. Unlike other food freshness monitoring approaches that require a thin film battery for operation of an RFID sensor and fabrication of custom-made sensors, our developed passive RFID sensing approach combines advantages of both battery-free and cost-effective sensor design and offers response selectivity that is impossible to achieve with other individual sensors.

Keywords

passive RFID sensors; multivariate data analysis; food safety; food freshness; bacteria detection; bacerial growth; milk; fish; *E. Coli*

Introduction

Radio frequency identification (RFID) sensors are finding their diverse applications when an unobtrusive sensor form factor, battery-free design, and minimal sensor cost are the top three requirements for a new sensor. Examples of diverse applications include pharmaceutical, warehousing, agricultural, industrial, food safety, and security.[1,2] Benefits of RFID sensors for food quality and safety, as compared to tethered sensors, include the non-obtrusive nature of their installations, higher nodal densities, and lower installation costs without the need for extensive wiring.[3–5] In addition, a significant advantage of RFID[6] and other electronic sensors[7–12] over optical sensors[13–15] is in the ability to perform measurements through non-transparent packaging.

There are several developed battery-free wireless sensing technologies based on magnetoelastic,[16] thickness shear mode,[17–19] surface acoustic wave,[20,21] magnetic acoustic resonance,[22,23] and resonant LCR (inductor-capacitor-resistor)[7,24] transducers. Several

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approaches for battery-free RFID sensing have been explored, e.g. based on chipless RFID sensors.[12,25,26]

We recently developed a methodology to implement passive RFID tags for physical, chemical, and biological sensing.[5,27–35] In our RFID sensing approach, the resonance impedance spectrum of the sensor antenna is measured and further correlated with chemical, biological, or physical properties of the environment. This correlation is performed using multivariable response of the RFID sensor computed from the measured impedance spectrum.

The complementary driving forces in successful sensor development are innovative ideas and the market size for new sensors. The market size is often but not always is supported by the regulatory requirements. If both driving forces are strong, the sensor development moves from its initial proof-of-concept technology readiness level to the commercialization of the sensor technology.[5]

The sizes of markets for food safety testing products ($0.25 B) and pathogen detecting sensors ($0.5 B)[36] and provide exciting opportunities for the development of new sensing technologies for food quality and safety. Intelligent labeling of food products to indicate and report their freshness and other conditions is one of important possible applications of the developed RFID sensors. Unlike other food freshness monitoring approaches that require a thin film battery for operation of an RFID sensor[6] and fabrication of custom-made sensors,[8,10,37] our developed passive RFID sensing approach combines advantages of both battery-free and cost-effective sensor design and offers response selectivity that is impossible to achieve with other individual sensors.[27,29,31]

In this review, we summarize result on the development of RFID sensors for food quality and safety. In these sensors, the electric field generated in the RFID sensor antenna extends out from the plane of the RFID sensor and is affected by the ambient environment providing the opportunity for sensing. This environment may be in the form of a food sample within the electric field of the sensing region or a sensing film deposited onto the sensor antenna. Examples of applications include monitoring of freshness of milk, freshness of fish, and bacterial growth.

General principles of design and operation of RFID food sensors

In order to assess the broad applicability of the developed sensors for food safety applications, it is critical to understand the general principles of their design and operation (see Figure 1). The equivalent circuit of the developed sensors forms an inductor-capacitor-resistor (LCR) circuit and is described by the inductance L_A, capacitance C_A, and resistance R_A of the sensing antenna coil, capacitance C_S and resistance R_S of the sensing region, and capacitance C_C and resistance R_C of the integrated circuit (IC) chip (see Figure 1A). Reading and writing of digital information into the RFID sensor and measurement of impedance of the RFID sensor antenna are performed via mutual inductance coupling between the RFID sensor antenna and the pickup coil of a digital/analog sensor reader.

Impedance spectra $\check{Z}(f)$ of the sensor are measured using a laboratory or a portable network analyzer component and digital data from an IC chip is measured with a digital RFID reader component[29] of our custom sensor reader. Digital data include sensor calibrations, food manufacturing data, end-user data, etc. The network analyzers are used to scan the frequencies over the range of interest (typically centered at 13 MHz with a scan range of ~10 MHz).

The electric field generated in the RFID sensor antenna extends out from the plane of the RFID sensor (Figure 1B) and is affected by the ambient environment providing the opportunity for sensing. This environment may be in the form of a food sample within the electric field of the sensing region or a sensing film deposited onto the sensor antenna. In both cases, the impedance of the antenna circuit $\check{Z}(f)$ is modulated through the changes in capacitance C_S and resistance R_S of the sensing region. This sensing region can be in the form of a full antenna or a complementary region in contact with the antenna.[32] Numerous types of sensing materials applicable for the food quality sensing were recently analyzed.[5]

To achieve accurate and precise measurements using our sensors, we measure the real $Z_{re}(f)$ and imaginary $Z_{im}(f)$ parts of the impedance spectra $\check{Z}(f)$ and calculate several spectral parameters. A schematic representation of the real $Z_{re}(f)$ and imaginary $Z_{im}(f)$ parts of the impedance spectrum $\check{Z}(f)$ of the sensor without possible effects from a pick up coil is illustrated in Figure 1C. Several calculated spectral parameters include the frequency position F_p and magnitude Z_p of $Z_{re}(f)$ and the resonant F_1 and antiresonant F_2 frequencies of $Z_{im}(f)$. Additional parameters can also be calculated (impedance magnitudes Z_1 and Z_2 at F_1 and F_2 frequencies, respectively, zero-reactance frequency, quality factor, etc). From the measured parameters, resistance, capacitance, and other parameters of the resonant antenna can be also determined. Figure 2 shows examples of RFID sensors applied in our studies for food quality and safety.

Uncontrolled temperature fluctuations produce independent effects on the different components of the equivalent circuit. These independent effects are correlated with the spectral features of the resonance impedance spectra and are resolved by the multivariable response of the sensor.[33,38]

For scenarios when the food is irradiated by ionizing radiation as a food safety measure to destroy bacteria, pathogens, and pests,[39,40] conventional RFID IC memory chips do not survive the applied radiation dose that can be up to 30 kGy. We have developed a technical solution to solve this problem where an IC chip is based on the Ferroelectric Random Access Memory (FRAM) technology and provides reliable gamma-resistant RFID tags and sensors.[41] The FRAM memory chips have 2000 bytes of user memory (MB89R118A, Fujitsu Microelectronics Ltd, Japan)[42] and are made using a standard RF signal modulation circuitry fabricated using a 0.35-μm complementary metal-oxide semiconductor (CMOS) process and a non-volatile FRAM memory.[43] A photo of this IC chip is shown in Figure 3A while one of our RFID sensors with such an IC chip is shown in Figure 3B.

Examples of applications

Monitoring of milk freshness

Milk is a major component of diverse diets across the world and has been the supply source, especially in the developing world, of a wide range of nutrients.[44] Milk spoilage is a result of growth of micro-organisms that modifies the composition of the medium with the metabolic products, changes the ionic content and conductivity of milk.[45] Hence, determination of milk quality is critical in order to maintain food safety and human health. Measured dielectric and viscosity parameters of milk have been successfully related to the milk freshness and milk adulteration.[10,44,46–48] Noninvasive and noncontact determination of milk quality attracts significant research and commercial interest.[10,37,46]

Our RFID sensors for monitoring of freshness of milk were constructed using 23 × 38 mm RFID tags from Texas Instruments (Plano, TX). Changes in the dielectric properties of milk were sensed with these RFID sensors that had an adhesive backing that has been attached to the side wall of the milk cartons. Figure 4A illustrates a schematic of the sensor positioning on milk carton and sensor-response readout with a pick-up coil. Two types of milk were used for evaluation, fat-free milk and whole milk. Before the RFID sensing experiment, reference measurements of the dielectric properties of fresh and spoiled milk were performed.

To determine milk spoilage rate, which is related to the changes in the dielectric properties of milk during storage, non-invasive determinations with RFID sensors were done directly through the walls of original milk cartons. Figure 4B depicts a photo of milk cartons with attached RFID sensors and demonstrates the simplicity of this sensing technology. A control experiment was performed with an RFID sensor attached to a carton filled with water that was identical to that filled with milk. Sensors monitored the change in solution dielectric constant as a function of storage time by taking advantage of the electromagnetic field penetration depth out of plane relative to the sensors and performing analysis directly through the original thin wall of the milk cartons.

Results of real time non-invasive monitoring of the condition of the two types of milk in cartons and the control water sample at room temperature are presented in Figure 5. The data illustrates different rates of spoilage of whole and fat free milk and no signal change for the control RFID sensor. The change in F_p signal of the RFID sensor due to changes in the dielectric property of milk was indicative of milk spoilage and was correlated with results obtained from the reference measurements, before and after milk spoilage. We also observed a small drop in the sensor response during an initial stage of milk monitoring. This behavior was observed in the past with impedance probes inserted in milk[45,49] and was attributed to being the result of several competing mechanisms of milk spoilage.

Automatic determinations of milk freshness were further performed (see Figure 6). For these measurements, 47 × 47 mm RFID tags from Texas Instruments (Plano, TX) were employed. Measurements of the resonant properties of the sensors were initially performed manually at different sensor positions relative to the pick-up coil. Further, a program, written in LabVIEW automatically determined the resonant properties of the sensors without the positioning effects of sensors relative to the pick-up coil and discriminated between cartons with fresh and spoiled milk.

Monitoring of fish condition

Using RFID sensors, food freshness can be determined not only by monitoring of changes in dielectric properties of individual food items, but also by adding enhanced functionality through the use of specific sensing materials as components of the sensors. By using such sensing materials, detection of food spoilage can be performed on a much shorter time scale. Detection of volatiles from food is a well-established method for determination of food freshness and spoilage.[50] The use of high-end laboratory instrumentation provides new insights on the evolution of diverse individual volatiles during food storage and determines food freshness and spoilage “markers”.[50,51] These markers can be monitored using sensor arrays (electronic noses)[52–56] or using more non-obtrusive and ubiquitous devices.[5,8,12] These monitoring devices can become a part of smart packaging[15,57] or can be smart food labels.[12]

Determination of fish freshness and spoilage based on measurements of characteristic odor volatiles is one of the main directions of the development of smart packaging because of the significant demand and market value of different fish products.[50] Volatiles contributing to odor of newly caught fresh fish originate from the diverse microflora and include C6 – C9 alcohols and carbonyl compounds, while volatiles that contribute to odor of fish during spoilage include ethanol, trimethylamine, ammonia, hydrogen sulphide, and some others which are the results of microbial spoilage.[50] Volatiles responsible for the fishy odor are the result of bacterial metabolism and include ammonia and volatile amines known as total volatile basic nitrogen (TVB-N) compounds.[57] Sensitive and selective detection of the TVB-N compounds is an active area of research.[57–61]

RFID sensors for monitoring of fish freshness were constructed from available 47 × 47 mm, RFID tags from Avery Dennison (model AD 709, Flowery Branch, Georgia) by draw-coating a polyaniline polymer to form a sensing film onto the antenna. Polyaniline is one of conjugated polymers also known as intrinsically conducting polymers. Conjugated polymers exhibit several mechanisms of molecular gas recognition including changes in density and charge carrier mobility, polymer swelling, and conformational transitions of polymer chains.[62,63] The strong acid-base interactions between the TVB-N compounds and the polyaniline polymeric film produce strong change in the film conductivity and lead to the high sensor sensitivity.

Initially, these RFID sensors were tested for the detection of ammonia gas and demonstrated a 500 part-per-trillion (ppt) detection limit (see Figure 7A). This achieved low detection limit was very attractive for the application of these sensors for the monitoring of fish freshness.[5] Further, the formulation of the polyaniline-based sensing film was optimized to minimize effects of water vapor (see Figure 7B). This reduced sensitivity to water vapor was possible due to the strong irreversible deprotonation chemical reaction of polyaniline in the presence of ammonia and only weak hydrogen bonding reversible interactions with water vapor.

For the evaluation of response of RFID sensors to fish odor during storage, five RFID sensors were arranged in separate petri dishes with ~ 200-mL headspace and were monitored at once using a multiplexed sensor reader. Three petri dishes contained individual ~ 20 g samples of salmon filet on a water-soaked liner. Two other sensors served as negative controls. The first control sensor was in a dry petri dish. The second control sensor was in a petri dish that contained only a water-soaked liner. Sensor positioning into the headspace of the petri dish with the fish sample and sensor-response readout with a pick-up coil are illustrated in Figure 8.

Results of experiments monitoring fish freshness at room temperature are presented in Figure 9. The negative control sensors exposed to low humidity and 100 %RH conditions demonstrated no increase in sensor impedance. Amines that were generated during salmon storage produced a significant increase in sensor impedance due to the deprotonation of polyaniline as detected in ~ 2 h with these sensors. Optical sensors based on colorimetric pH reagents immobilized onto solid sensing films also have been used for detection of ammonia and amines, however with higher detection limits (mid-range ppb or higher)[57,59,64–66] as compared to the 500-ppt detection limit achieved with our developed RFID sensors.

Direct monitoring of bacteria growth

Direct monitoring of bacterial growth is important for food safety applications. Traditionally, quantitation of bacterial contamination is performed using automated bacterial counting and based on analysis of markers of bacterial metabolism.[67,68] Additionally, detection of bacteria has been carried out using sensors and sensor arrays.[69,70] Sensitive and selective detection of bacteria and its growth in a non-invasive, non-obtrusive non-contact fashion using wireless or proximity sensors is currently an active area of research.[12,71,72]

We have demonstrated the applicability of developed RFID sensors for monitoring of bacterial growth using *E. coli* as a model system. Monitoring of bacteria was performed using 23 × 38 mm RFID tags from Texas Instruments (Plano, TX). These tags were employed as RFID sensors by measurements the resonance impedance of the antenna circuit. Two RFID sensors were positioned into petri dishes and were kept in an incubator at 37 °C. The RFID sensors were integrated with a food wrap film as detailed in Figure 10A. Each sensor was attached with its original adhesive to one side of the food wrap film while the other side of the food wrap film was exposed to a solution. One of the RFID sensors (sensor 1) was associated with a solution containing *E. coli*, while another sensor (sensor 2) was associated with a sterile, bacteria-free solution. Reading of the resonance impedance of the sensors was performed using a pickup coil from the bottom of the petri dish and RFID sensor as shown in Figure 10A. A photo of sensor 1 (with *E. coli*-containing solution) and sensor 2 (with control bacteria-free sterile solution) in the incubator is shown in Figure 10B. An initial ~ 1.5 h time period (not shown) was a stabilization period where responses of sensors 1 and 2 equilibrated. This initial time may be associated with the equilibration of liquid distribution between the solution and the food wrap.

Upon the equilibration, the sensor 1 started to demonstrate an increase in the sensor signal while the response of the control sensor 2 remained stable. Figure 11 illustrates representative ΔF_p and ΔZ_p results of this experiment. The increase in the response of sensor 1 was associated with the change in the dielectric contact of the food wrap layer due to the accumulation of bacteria in the sensing region, while the constant response of sensor 2 was associated with no change in the dielectric contact of the food wrap layer due to the exposure to the sterile solution.

Responses of sensor 1 and sensor 2 were further analyzed using a pattern recognition technique to quantitatively determine the differences in the responses between sensors exposed to bacteria and to sterile conditions. As a pattern recognition technique, we employed principal components analysis (PCA), a robust unsupervised pattern recognition tool.[73] PCA is a multivariate data analysis tool that projects the data set onto a subspace of lower dimensionality with removed collinearity. PCA achieves this objective by explaining the variance of the data matrix in terms of the weighted sums of the original variables F_p, F_1, F_2, and Z_p or complete $Z_{re}(f)$ and $Z_{im}(f)$ of impedance spectra with no significant loss of information. These weighted sums of the original variables are called principal components (PCs). Results of PCA are presented in Figure 12 illustrating that the RFID sensors provided the robust ability to discriminate between bacterial growth and sterile conditions.

Future developments

This study demonstrated the applicability of our RFID sensors for the monitoring different aspects of food quality including freshness, ageing, and spoilage. In contrast to known wireless sensors, our developed RFID sensors combine several measured parameters from the resonant sensor antenna with multivariate data analysis and deliver unique capability of sensing with rejection of environmental interferences with a single sensor. Similar to our other RFID sensors that have the ability for self-correction for temperature effects,[38,74] our bacterial RFID sensors reported here also have this self-correction ability for temperature instabilities. Overall, to be accepted for diverse practical application scenarios, RFID sensors should have the ability for the self-calibration and for the self-correction for variable ambient conditions (temperature, repositioning, and others). Future steps are being planned for field-testing of these sensors in numerous conditions.

The application of battery free (passive) 13.56 MHz RFID tags as wireless or proximity sensors is attractive where the high quality of sensor performance is needed at low cost and when battery-free operation is critical. Our sensors, when integrated into single-use packaging components can provide several important capabilities previously unavailable from other single-use sensing technologies: (1) common sensor platform for measurements of physical, chemical, and biological parameters, (2) multi-parameter monitoring with individual sensors, and (3) simultaneous digital identification functionality that provides information about the packaging component and the calibration coefficients of the RFID sensor.

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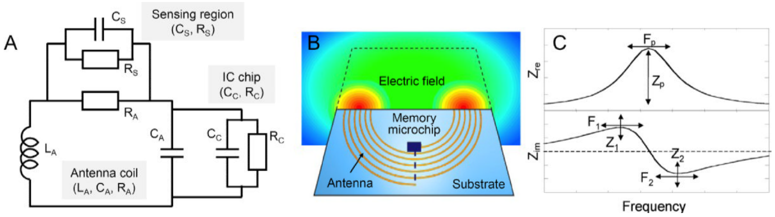

Figure 1.

Operation principle of developed passive RFID sensors. (A) Sensor equivalent circuit described by the inductance L_A, capacitance C_A, and resistance R_A of the sensing antenna coil, capacitance C_S and resistance R_S of the sensing region, and capacitance C_C and resistance R_C of the IC chip. (B) Schematic representation of the origin of response of RFID sensors to parameters of interest. (C) Measured impedance spectrum (real part and imaginary part of impedance) and examples of parameters for multivariate analysis (see text for details).

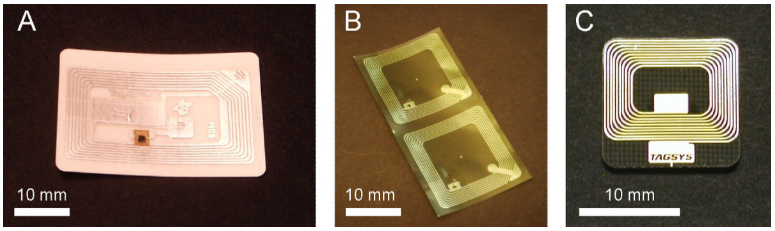

Figure 2.

Examples of employed RFID sensors based on (A) Texas Instruments RFID tag, (B) Avery Dennison RFID tag, (C) TagSys RFID tag.

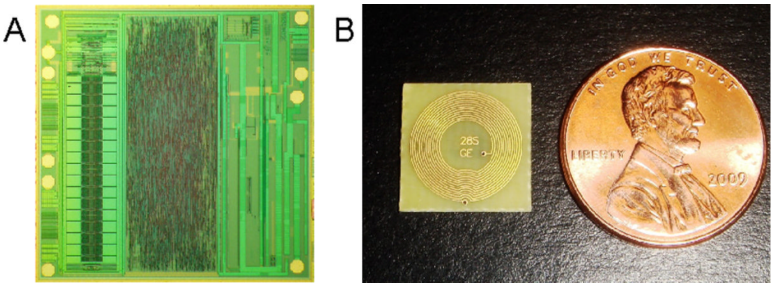

Figure 3.
Photographs of (A) FRAM IC memory chip MB89R118A and (B) Developed RFID sensor for gamma-sterilizable applications. Sensor diameter = 10 mm.

Figure 4.

RFID sensor layout for demonstration of determination of milk freshness. (A) Schematic of sensor positioning onto a milk carton and sensor-response readout with a pick-up coil. (B) Photo of milk cartons with attached RFID sensors.

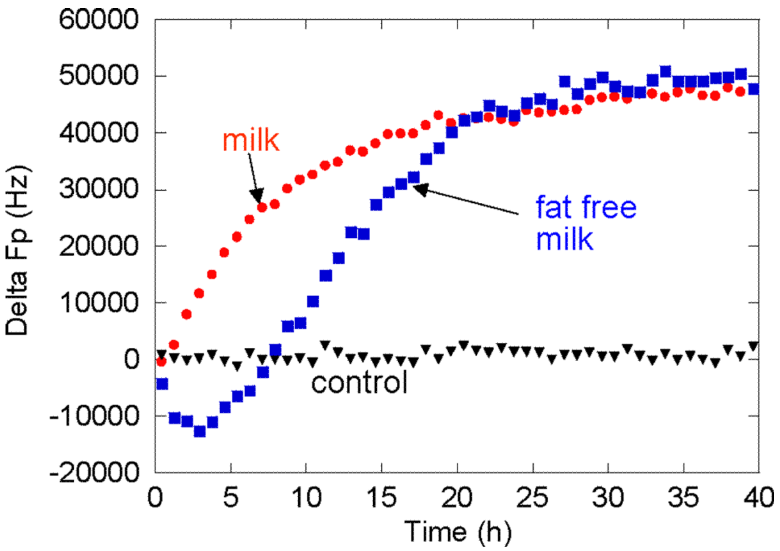

Figure 5.

Non-invasive monitoring of whole and fat-free milk using disposable RFID sensors. Response of RFID sensors to spoilage of two types of milk and to a control (water) sample[29].

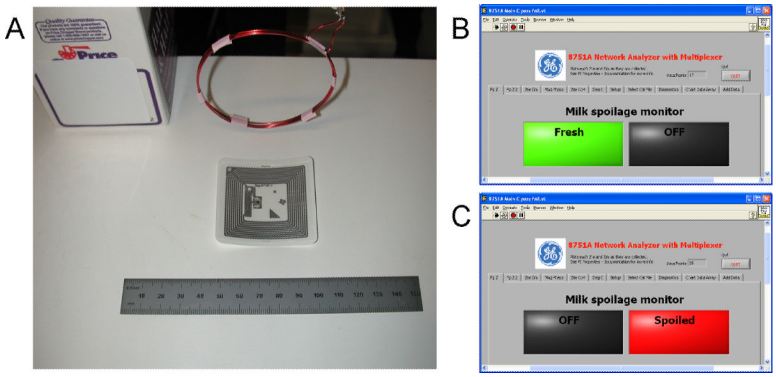

Figure 6.

Automatic non-invasive determination of milk freshness. (A) A milk carton with an attached RFID sensor and an associated pick-up coil. (B, C). Front panel of written program for an automatic determination of the resonant properties of the sensors without the positioning effects of sensors relative to the pick-up coil. The program was able to discriminate between cartons with fresh and spoiled milk.

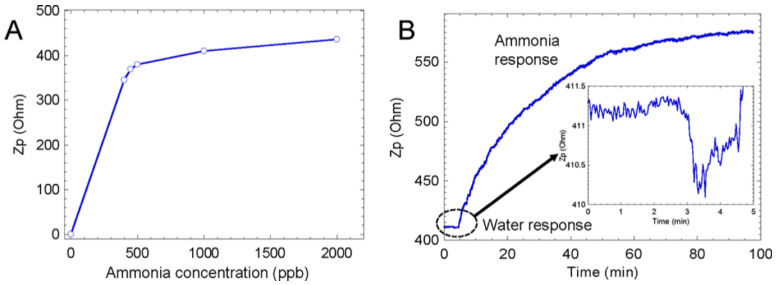

Figure 7.

Demonstration of high sensitivity and selectivity of developed RFID sensors for fish freshness. (A) Calibration curve of RFID sensor for ammonia detection. (B) Response of the sensor to water vapor (~ 75% relative humidity) and to 500 ppb of ammonia.

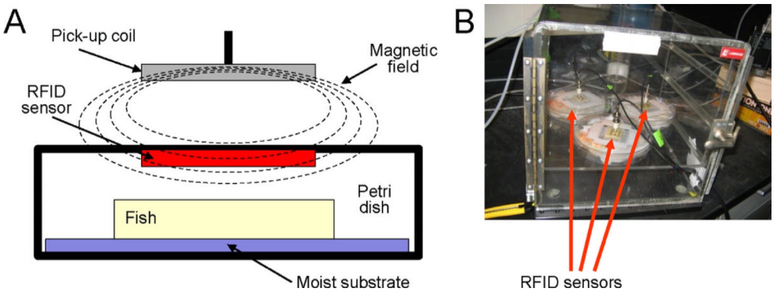

Figure 8.
RFID sensor layout for demonstration of monitoring of fish freshness. (A) Schematic of sensor positioning into the headspace of a plastic container with a fish sample and sensor-response readout with a pick-up coil. (B) Photo of RFID sensors attached to plastic containers with fish samples.

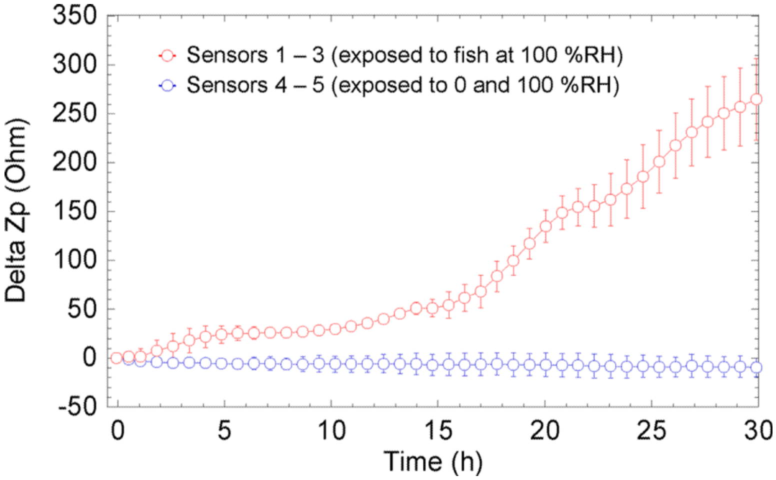

Figure 9.

Monitoring of fish freshness using polyaniline-based RFID sensors. Sensors 1 – 3 were positioned into the headspace of a petri dish with individual ~ 20 g samples of salmon filet on a water-soaked liner. Sensor 4 served as the first negative control positioned in a low humidity headspace. Sensor 5 served as the second negative control positioned in a headspace only with a water-soaked liner. Sensors 1 – 5 were monitored simultaneously at room temperature using a sensor multiplexer after 1 h of equilibration time to reach the state-state condition in all sensors. Results are plotted as means and SD for sensors 1 – 3 (red trace) and 4 – 5 (blue trace)[5].

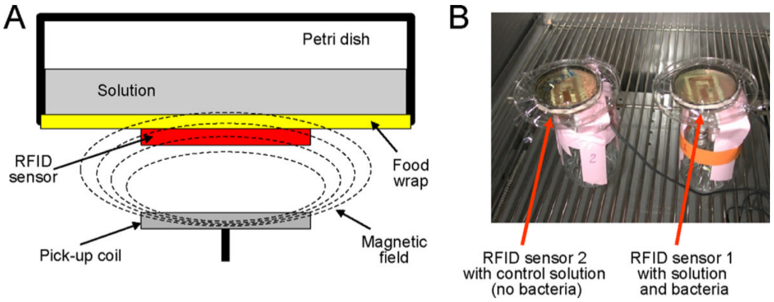

Figure 10.

RFID sensor layout for demonstration of monitoring of bacterial growth. (A) Schematic of sensor positioning into a plastic container. (B) Photo of two RFID sensors in the incubator for demonstration of monitoring of bacterial growth.

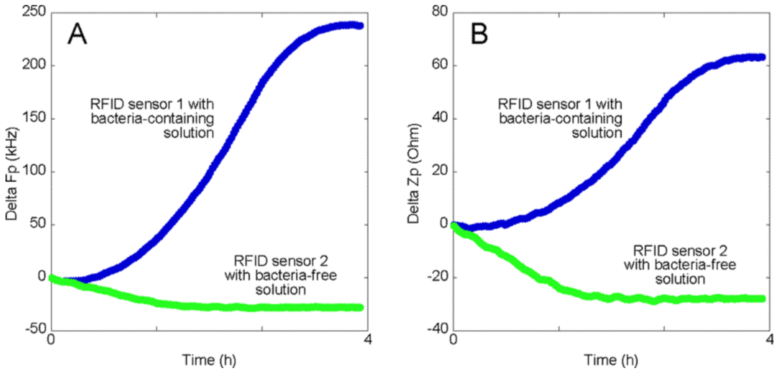

Figure 11.

Real-time monitoring results using two RFID sensors. Sensor 1 was with a solution containing *E. coli* bacteria; Sensor 2 was with a sterile bacteria-free solution. (A) and (B) are representative results of ΔF_p and ΔZ_p responses, respectively.

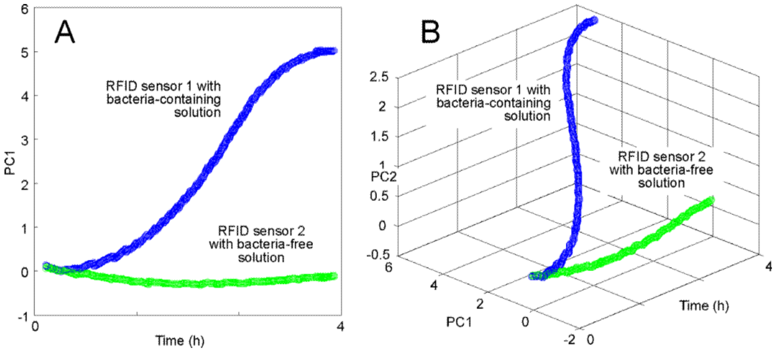

Figure 12.

Results of pattern recognition analysis of a bacterial monitoring process demonstrating pattern discrimination between RFID sensors responses in the presence (sensor 1) and absence (sensor 2) of *E. coli* bacteria. (A) Results of PC1 response vs. time. (B) Results of PC1 response vs. PC2 response vs. time.