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A novel microwave sensor to determine particulate blend composition on-line

John Austin, Anshu Gupta, Ryan McDonnell, Gintaras V. Reklaitis, Michael T. Harris*

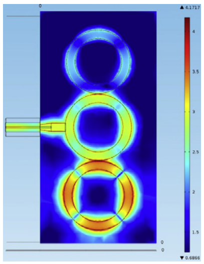
Purdue University, School of Chemical Engineering, Forney Hall of Chemical Engineering, Room 3007, 480 Stadium Mall Drive, West Lafayette, Indiana, 47907-2100, United States

HIGHLIGHTS

- A novel microwave sensor was designed to measure chemical composition.
- This sensor and an NIR probe were used to monitor a flowing pharmaceutical blend.
- The microwave sensor had a comparable accuracy to the NIR probe.

GRAPHICAL ABSTRACT

COMSOL simulation results showing how the norm of the electric field varies along the sensor surface when the bottom ring resonates at approximately 4.3 GHz. The color-coding shown is on a log base 10 scale. Thus, the field strength around the top ring is less than 1% of the strength in the bottom ring, indicating that the rings can resonate independently of one another. The sensor designed is capable of measuring chemical composition and moisture content of particulate blends in-line.



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ABSTRACT

Due to the ease with which particulate blends tend to segregate, blend uniformity and chemical composition are two critical control parameters in nearly all solids manufacturing industries. The prevailing wisdom has been that microwave sensors are not capable of or sensitive enough to measure the relative concentrations of components in a blend. Consequently, it is common to turn to near infrared sensing to determine material composition on-line. In this study, a novel microwave sensor was designed and utilized to determine, separately, the concentrations of different components in a blend of microcrystalline cellulose, acetaminophen, and water. This custom microwave sensor was shown to have comparable accuracy to a commercial NIR probe for both chemical composition and moisture content determination.

1. Introduction

Rapid and accurate determination of process variables is valuable in nearly every manufacturing industry. In those that deal with the manufacture of particulates, where segregation is always a concern, chemical composition is one of the most important process and quality control variables. For instance, in the manufacture of pharmaceutical tablets, small changes in composition can mean

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* Corresponding author at: Purdue University Neil Armstrong Hall of Engineering, G051 701 West Stadium Avenue West Lafayette, IN, 47907 United States. Tel.: +1 765 494 0963; fax: +1 765 494 0805.

E-mail addresses: austin6@purdue.edu (J. Austin), mtharris@purdue.edu (M.T. Harris).

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the difference between an ineffective dose, an efficacious one, or a toxic one.

Several techniques have been investigated as a means of monitoring the chemical composition of a particulate blend. Through the use of multivariate statistics, nuclear magnetic resonance has been used to characterize complex mixtures [1]. Hyperspectral imaging has been used to monitor the composition of manufactured food products for quality and safety control [2]. Solid phase molecular fluorescence spectroscopy has also been used to determine the chemical composition of complex mixtures with minimal sample preparation and low cost [3,4]. However, none of these techniques has shared the success of near infrared (NIR) spectroscopy. NIR has become the method of choice to determine material composition on-line [5,6].

Despite its popularity, NIR sensing of particulates suffers from several significant drawbacks. Most importantly, NIR radiation can only penetrate into a material a very short distance, on the order of millimeters [7]. This is not a significant drawback in liquid systems, where solutions can often be assumed well mixed and thus homogenous. Particulate blends can segregate as a result of many different phenomena, most notably due to differences in particle size and cohesivity [8]. With a penetration depth of only a few millimeters, segregation can render NIR results to be inaccurate. Other notable drawbacks of NIR spectroscopy include sensitivity to physical properties, such as particle size and roughness, and the necessity to develop models using complex chemometric software.

Microwave sensors do not suffer from these drawbacks. First, microwave sensors are able to monitor bulk material properties due to their increased penetration depths [9]. Second, microwave sensors are able to monitor moisture contents with great accuracies, due to large differences in dielectric properties between solids and polar liquids. Commonly, empirical and semi-empirical linear regression models are employed to calibrate microwave sensors. Therefore, complex chemometric software is usually not needed. Microwave interactions with particulate mixtures can be complex and are often a function of at least frequency, temperature, moisture content, crystallinity, and composition. Due to the highly nonlinear behavior of some systems, artificial neural networks have also been employed to monitor particulate properties with high levels of accuracy [10].

Microwave rotational spectrometers have been used off-line in laboratories for the determination of chemical composition of gases [11]. Often, these off-line sensors make use of highly sensitive Fabry-Perot resonators. The design and use of these resonators requires that they be very precisely controlled and are thus not suitable for on-line analysis during particulate manufacture. To the authors' knowledge, microwave sensors have not been used on-line to determine the chemical composition of a particulate product. This study seeks to demonstrate that microwave sensors can be viable measurement devices for determining the chemical composition of a particulate blend on-line.

2. Methods

2.1. Development of microwave sensor

To detect material composition on-line, a novel microwave sensor was designed and developed. Most resonant microwave sensors operate around a single resonance, each providing two independent variables to the user. For instance, many sensors are calibrated to provide both the real and imaginary components of the effective dielectric constant at the frequency of resonance [12]. To discern the concentrations in a system with several components, one needs more independent variables, and thus the microwave sensor designed needs to have multiple resonances. Ideally, microwave sensors used to determine chemical composition should have resonances spread out over the available range of frequencies, since the dielectric properties of a material do not normally differ significantly with small changes in frequency. In addition, it is necessary for the components in the material under test to have noticeably different valued dielectric properties in the microwave region. For instance, it would be very difficult to tell the difference between two similar polymers or between polymers based on the same unit, but of different chain lengths.

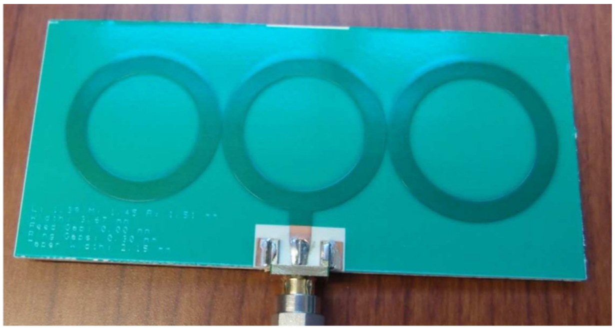

Fig. 1. Top view of planar sensor as fabricated.

A sensor with six resonances over the 2–8 GHz region was developed using the finite element package COMSOL Multiphysics, available from COMSOL Inc. (Burlington, MA). As shown in Fig. 1, the final sensor design consisted of three parallel ring resonators made from 62 mil Rogers 4350 with 1 oz copper by Advanced Circuits (Aurora, CO). To match the impedance of the 50-ohm coaxial feed cable, the microstrip lines were manufactured to be 3.67 mm wide. A solder mask (PSR-4000BN from Taiyo America, Inc.; Carson City, NV) was applied to the sensor surface to make it more rugged and to reduce the risk of material sticking to the surface. As reported by the vendor, the solder mask had an approximate thickness of 25 μm. Due to this negligible thickness, the solder mask was not simulated in COMSOL Multiphysics to significantly reduce computation time.

The sensor was designed with three parallel ring resonators for several reasons. Firstly, as was discovered from simulations, this design allows the quality factors of the resonances to be very high. The accuracy and sensitivity of a resonant sensor both increase with its quality factor [13]; it is a measure of the energy stored in a resonator to the energy dissipated per cycle. A standard design using a single ring resonator would normally yield a quality factor in the range of 100–300. In this design, the middle ring couples to the outer rings magnetically and allows them to reach quality factors in the thousands. Thus, this design allows for significantly more accurate sensing.

Secondly, it allows material properties to be determined uniquely from either side of the sensor, allowing the user to determine if segregation is occurring. By slightly varying the radii of the three rings, they can be made to resonate independently of one another. The outer diameters of the three rings were, from left to right (Fig. 1): 2.78 cm, 2.9 cm, and 3.02 cm. These diameters were chosen based on trial and error from simulations and produced the largest quality factors while maintaining unique resonances. A larger difference in the diameters would result in less defined resonances while a smaller difference would allow the rings to resonate together. How the norm of the electric field varies at resonance can be seen in Fig. 2. Keeping in mind that the color-coding is on a log base 10 scale, one can observe that the bottom ring is able to resonate independently of the top ring, with more than 100× the electric field strength around the bottom ring than around the top ring.

Furthermore, ring resonators were chosen because they have both higher quality factors and are more compact than their linear,