Temperature and moisture dependent dielectric properties of legume flour associated with dielectric heating

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\textbf{Abstract}

Dielectric properties data are important in developing thermal treatments using radio frequency (RF) and microwave (MW) energy and are essential in estimating heating uniformity in electromagnetic fields. Dielectric properties of flour samples from four legumes (chickpea, green pea, lentil, and soybean) at four different moisture contents were measured with an open-ended coaxial probe and impedance analyzer at frequencies of 10–1800 MHz and temperatures of 20–90°C. The dielectric constant and loss factor of the legume samples decreased with increasing frequency but increased with increasing temperature and moisture content. At low frequencies and high temperatures and moisture contents, negative linear correlations were observed between the loss factor and frequency on a log-log plot, which was mainly caused by the ionic conductance. At 1800 MHz, the dielectric properties data could be used to estimate the legume sample density judging from high linear correlations. Loss factors for the four legume samples were similar at 27 MHz, 20°C and low moisture contents (e.g. <15 g/100 g). At the highest moisture content (e.g. 20 g/100 g) soybean had the highest loss factor at 27 MHz and 20°C, followed by lentil, green pea, and chickpea. The difference in loss factor among the four legumes did not show clear patterns at 915 MHz. Deep penetration depths at 27 MHz could help in developing large-scale industrial RF treatments for postharvest insect control or other applications that require bulk heating in legumes with acceptable heating uniformity and throughputs.

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1. Introduction

Chickpea (Cicer arietinum), green pea (Pisum sativum), lentil (Lens culinaris), and soybean (Glycine max) are four important rotational legumes in the United States. Their production reached 69,870, 599,416, 147,278, and 87,196,000 metric tons, respectively, in 2006 with a value of about US$38, 72, 36, and 18,922 million, respectively (USDA-NASS, 2007). About 31, 72, 89, and 36 kg/100 kg of their total productions are for export to India, Spain, and Latin American countries such as Colombia, Cuba, and Peru (USADPLC, 2007). A major problem in production, storage and marketing of these legumes is infestation by insect pests (USADPLC, 2007). Importing countries may set both general and specific conditions concerning pests. These may include phytosanitary disinestation treatments such as fumigation or certification that the shipment is free of certain insects.

Consumer and environmental concerns over the use of chemical fumigants (UNEP, 1995) has generated interest in non-chemical alternatives. Radio frequency (RF) and microwave (MW) treatments have been studied as a non-chemical alternative for postharvest insect control in dried agricultural commodities, such as grain (Nelson, 1996), rice (Lagunas-Solar et al., 2007; Zhao, Qiu, Xiong, & Cheng, 2007), walnut (Wang, Monzon, Johnson, Mitcham, & Tang, 2007a, 2007b), and wheat (Halverson, Burkholder, Bigelow, Nordheim, & Misenheimer, 1996). To develop potential phytosanitary RF and MW thermal treatments for legumes, it is important to determine the dielectric properties that govern the interaction between the electromagnetic energy and the legumes.

Dielectric properties of a product provide general guidance for selecting the optimal frequency range and bed thickness for uniform RF treatments (Wang, Tang, Johnson, et al., 2003). Many techniques have been developed for measurement of dielectric properties of different materials. The open-ended coaxial probe method is currently one of the most popular techniques for...
measuring complex dielectric permittivity of many materials. It has broad frequency bands, is easy to use, and does not need special sample preparations (Sheen & Woodhead, 1999; Wang, Tang, Cavallieri, & Davis, 2003). The well-developed theory makes it possible to obtain sufficiently accurate results both for medium-loss and high-loss materials, such as liquids (Guo, Trabelsi, Nelson, & Jones, 2007; Tanaka, Morita, Mallikarjunan, Hung, & Ezeike, 2002), foods (Engelder & Buffier, 1991; Içier & Baysal, 2004; Ryyânänen, 1995; Wang, Tang, Raszko, Kong, & Wang, 2008b), and fresh fruits (Guo, Nelson, Trabelsi, & Kays, 2007; Ikediala, Tang, Drake, & Neven, 2000; Nelson, 2005; Wang et al., 2005; Wang, Tang, Johnson, et al., 2003) as affected by temperature and moisture content (Guo, Tiwari, Tang, & Wang, 2008; Nelson, 1981; Wang et al., 2008b). However, it is difficult to use this method to measure the dielectric properties of low moisture legumes, because it requires close contact between the flat tip of the probe and irregularly shaped seeds. The measurement errors are reduced by using a ground sample of the seeds (Guo et al., 2008; Nelson, 1984; Nelson & Trabelsi, 2006). This allows relatively reliable measurement of dielectric properties for dry products using open-ended coaxial probe methods (Guo et al., 2008). Since dielectric properties of particulate materials depend on sample density (Berbert, Queriroz, & Mesquita, 2002; Nelson, 1984), the density of the compressed samples should match the true kernel density of the seeds. Such property data can be useful to design the RF treatment protocols for disinfecting these seeds.

Much research has been conducted on dielectric properties of dry products, such as bean (Berbert et al., 2002), coffee (Berbert et al., 2001), flaxseeds (Sacilik, Tarimci, & Colak, 2006), and grain seeds or flour (Lawrence, Nelson, & Kraszewski, 1990; Nelson, 1984; Nelson, Soderholm, & Jung, 1953; Nelson & Trabelsi, 2006; Trabelsi, Kraszewski, & Nelson, 1998). Recently, Guo et al. (2008) reported dielectric properties of chickpea flour as influenced by frequency, temperature and moisture content. It is of interest to examine the effects of water activity on dielectric properties, to further estimate penetration depths, and to compare with the dielectric properties of other legumes.

The objectives of this study were 1) to determine the dielectric properties of green pea, lentil, and soybean flour over the frequency range of 10–1800 MHz, at temperatures from 20 to 90°C, and at four moisture content or water activity levels, 2) to compare the loss factors of the selected three legume flours with chickpea samples at the above frequencies, 3) to develop correlations between dielectric properties and the density of all four legume samples, and 4) to evaluate the penetration depth of the four legume samples to estimate the optimal thickness of treatment beds in RF and MW systems.

2. Materials and methods

2.1. Materials and sample preparation

Seeds of green pea, lentil, and soybean were purchased from a local grocery store in Pullman, WA, USA. The sample compositions of the legumes, including ash, calorie content, carbohydrate, fat, moisture, protein, and sodium, were analysed by Columbia Food Laboratories, Inc., Corbett, OR, USA. The standard methods used for each measurement are summarized in Table 1.

<table>
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<th>Composition</th>
<th>Chickpea</th>
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<th>Lentil</th>
<th>Soybean</th>
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<td>11.5</td>
<td>AOAC 975.03</td>
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2.2. Measurements of density, moisture content and water activity

Seed density of legumes at different moisture contents was measured by a liquid displacement method (Guo et al., 2008). Because toluene (C₇H₈) shows little tendency to soak into the sample, flows smoothly over the sample surface, and has stable specific gravity and viscosity, it was used as the displacing liquid. Seed density was determined by dividing the weight of randomly selected 25 g samples by the volume occupied by those seeds as measured with toluene in 100 ml pycnometers. For each sample, experiments were replicated three times and the mean density values were calculated from the replicated results.

Legume moisture content was determined by drying triplicate 2–3 g flour samples in aluminium moisture dishes in a vacuum oven (ADP-31, Yamato Scientific America Inc., Santa Clara, CA, USA) at 130°C for 1 h (AOAC, 2002). The samples were cooled in a desiccator with CaSO₄ before reweighing to determine water loss. Moisture content was calculated from the initial and final weights of the samples. The mean moisture content of the samples was obtained from three replicates. Because the moisture content of legume samples can be related to the water activity using the moisture sorption isotherm, the water activity of each sample was also measured at room temperature using an AquA lab water activity meter (Series 3, Decagon Devices Inc., Pullman, WA, USA) with an accuracy of ±0.003 aw. After calibrating the meter with a standard salt solution, 5 g of the sample was placed inside the measuring chamber and the head sensor was fitted to seal the chamber. Measurements were conducted in triplicate.

2.3. Dielectric properties measurements

Dielectric properties of the compressed legume flour samples were measured at 201 discrete frequencies between 10 MHz and 1800 MHz using the open-ended coaxial probe technique with an impedance analyzer (model 4291B, Hewlett-Packard, Santa Clara, CA, USA). This frequency range covers three U.S. Federal Communications Commission (FCC) allocated RF frequencies (13, 27, and...
40 MHz) and one MW frequency (915 MHz) for industrial heating applications. The upper limit frequency (1800 MHz) is close to another FCC allocated MW frequency (2450 MHz) applied for domestic MW ovens. Because thermal treatments for controlling insects in legumes are generally conducted at temperatures between 20 and 90 °C with moisture variations between 8 and 20 g/100 g w.b., the dielectric properties were measured over these temperature and moisture ranges. A detailed description of the system was previously published (Guo et al., 2008; Wang, Tang, Johnson, et al., 2003). The compressed sample was placed in a stainless steel sample cell (20 mm inner diameter with 30 mm length). About 0.3 g legume flour was added on the top of the compressed sample to ensure a close contact with the probe via a pressure spring. The sample moisture content was maintained during the measurements in the water and air tight test cell and the sample temperature was controlled by circulating water (15 L/min) from a water bath (model 1157, VWR Scientific Products, Niles, IL, USA) into the jacket of the test cell. A 50 g/100 g water and 50 g/100 g ethylene glycol solution was used as a heat transfer medium. A type-T thermocouple (0.8 mm diameter and 0.8 s response time) was used to monitor the sample temperature.

Before the dielectric properties measurement, the impedance analyzer was warmed up for at least 1 h and then calibrated with air, a short, a 50 Ω load, and a low-loss capacitor. The impedance analyzer and the coaxial probe were further calibrated with air, a short, a 50 Ω and a low-loss capacitor. The measurement accuracy of the system was about 5% for high-loss materials after following the standard calibration process. The measurement accuracy of the system was validated using butyl alcohol at 20 °C (Wang, Tang, Cavalieri, et al., 2003). Dielectric properties of each sample were measured at 20, 30, 40, 50, 60, 70, 80, and 90 °C. The temperature range was selected based on estimated insect tolerances (Wang, Ikediali, Tang, & Hansen, 2002) and lentil quality limitations (Tang & Sokhansanj, 1993). A frequency sweep took about 2 min. After each measurement, the water bath was adjusted to the next temperature level, with sample temperatures reaching the desired level in about 10 min. After each replicate, the probe and the sample cell were cleaned with deionized water and wiped dry. Dielectric properties data for legume flour samples were determined in duplicate. Mean values and standard deviations were calculated from the two replicates.

2.4. Effects of sample electrical conductivity and density on dielectric properties

Dielectric loss factors (\(\varepsilon''\)) are mainly dominated by ionic conduction (\(\varepsilon''_d\)) and dipole rotation (\(\varepsilon''_s\)) according to Rynnänen (1995):

\[
\varepsilon'' = \varepsilon''_d + \varepsilon''_s
\]

(1)

For liquid solutions or high moisture materials, Eq. (1) can be expanded into:

\[
\varepsilon'' = \varepsilon''_d + \frac{\sigma}{2\pi\varepsilon_0 f_0}
\]

(2)

where \(f\) is the frequency (Hz), \(\varepsilon_0\) is the permittivity of free space or vacuum (8.854 × 10⁻¹² F/m) and \(\sigma\) is the electrical conductivity (S/m). If the effect of dipole rotation is negligible at low frequencies (e.g. ≤200 MHz), after setting (\(\varepsilon''_s\)) = 0 and taking the logarithm on both sides, Eq. (2) becomes:

\[
\log \varepsilon'' = \log \left(\frac{\sigma}{2\pi\varepsilon_0 f_0}\right) - \log f
\]

(3)

Eq. (3) shows that a linear relationship between the loss factor and frequency can be established on a log-log plot with a slope of –1. In fact, using the above relationship, lost factors of several high moisture foods, such as mashed potato and salmon fillets, were estimated with good accuracy from electric conductivities directly measured by conductivity sensors (Guan, Cheng, Wang, & Tang, 2004; Wang et al., 2008b). But for solid materials of intermediate and low moisture contents, however, the slope was found to be greater than –1 (Guo et al., 2008; Liu, Tang, & Mao, 2009; Wang et al., 2005, 2008b). This suggests that Eq. (3) cannot be directly used for those materials. Thus, Liu et al. (2009) used a modified linear regression equation (\(\log \varepsilon'' = \alpha + \beta \log f\)) between the loss factor and frequency on a log-log plot and introduced the concept of effective electrical conductivity in the constant (\(\alpha\)). The intercept (\(\alpha\)) of the above relationship derived from loss factor data below 200 MHz was used to estimate the effective electrical conductivity (\(\sigma_e\)) of four legume flour samples as a function of temperature and moisture content (Liu et al., 2009) as follows:

\[
\sigma_e = 2\pi\varepsilon_0 10^\alpha
\]

(4)

High correlations have been reported between measured dielectric constant \(\varepsilon''\) and loss factor \(\varepsilon''\), each divided by the seed density, \(\rho\), for grains and chickpeas in the complex-plane plots in frequency range from 1800 to 18,000 MHz (Guo et al., 2008; Nelson & Trabelsi, 2006; Trabelsi, Kraszewski, & Nelson, 1997). This relationship has the following form:

\[
\frac{\varepsilon''}{\rho} = a \varepsilon'' - b
\]

(5)

where \(a\) and \(b\) are constants. Eq. (5) has been validated for selected microwave frequencies, e.g. 1800 MHz for chickpea and wheat, and 11300 and 18000 MHz for hard red winter wheat (Guo et al., 2008; Trabelsi et al., 1997). In order to assess the best frequency range for Eq. (5) in this study, the correlation between the measured \(\varepsilon''/\rho\) and \(\varepsilon''/\rho\) was compared for chickpea samples at four MW frequencies of 918, 1206, 1503 and 1800 MHz. Finally similar correlations were also determined for the four legume samples at 1800 MHz.

2.5. Penetration depth

Penetration depth of RF and MW power is defined as the depth where the power is reduced to 1/e (e = 2.718) of the power entering...
The penetration depth \( d_p \) in m of RF and MW energy in a material can be calculated according to von Hippel (1954):

\[
d_p = \frac{c}{2\pi f \sqrt{\epsilon \left( 1 + \left( \frac{\sigma}{\omega \epsilon_0} \right)^2 \right) - 1}}
\]

(6)

where \( c \) is the speed of light in free space \((3 \times 10^8 \text{ m/s})\). After obtaining the dielectric properties, the penetration depths of electromagnetic energy into the four legume samples were calculated at three frequencies (27, 915, and 1800 MHz), three temperatures (20, 60, and 90 °C) and all four moisture contents.

### 3. Results and discussion

#### 3.1. Dielectric properties as influenced by frequency, temperature and moisture

![Fig. 2. Moisture sorption isotherm of chickpea (□), green pea (○), lentil (△) and soybean (●) samples at 20 °C.](image)

![Fig. 3. Frequency dependence of the dielectric properties of compressed green pea flour samples at temperatures of 20 (□), 30 (○), 40 (△), 50 (×), 60 (+), 70 (−), 80 (○), and 90 °C (+) and the highest moisture content of 21.6 g/100 g.](image)

![Fig. 4. Frequency dependence of the dielectric properties of compressed lentil flour samples at temperatures of 20 (□), 30 (○), 40 (△), 50 (×), 60 (+), 70 (−), 80 (○), and 90 °C (+) and the highest moisture content of 21.5 g/100 g.](image)

Fig. 1 shows the seed densities of the four legume samples measured over a moisture range from 7.9 to 21.6 g/100 g. The seed density decreased with increasing moisture content. The mean seed density of soybeans was clearly less than that of the other three legumes. Generally, the trend of decreasing seed density with increasing moisture content was also reported for chickpea (Guo et al., 2008; Konak, Çarman, & Aydin, 2002), lentil (Amin, Hossain, & Roy, 2004; Tang & Sokhansanj, 1993), rice (Muramatsu, Tagawa, & Kasai, 2005), and soybean (Deshpande, Bal, & Ojha, 1993). This is probably due to the corresponding weight increase being less than the volume expansion as the moisture level increases.

Fig. 2 shows typical moisture sorption isotherms for the four legume flour samples at 20 °C. The moisture content increased with increasing water activity. Each moisture content level corresponded to a fixed water activity for chickpeas, green peas, lentils, and soybeans. Therefore, water activity dependent dielectric properties could be used to develop practical applications for rapid measurement of moisture content for legumes.

The log-log plot of the dielectric constant and loss factor at different frequencies is presented for compressed flour samples of green pea (Fig. 3), lentil (Fig. 4) and soybean (Fig. 5) over the...
temperature range from 20 to 90 °C at their highest moisture contents of 21.6, 21.5, and 19.9 g/100 g, respectively. Generally, both the dielectric constant and loss factor decreased with increasing frequency, especially at high temperatures. This is similar to observations made with chickpea samples (Guo et al., 2008). The dielectric constant and loss factor increased with increasing temperature, especially at RF frequencies. The loss factor had a negative relationship with frequency, especially at high temperatures and the low frequency range (e.g. below 200 MHz). This was because the ionic conductance played a dominant role under these conditions when the effect of dipole rotation was negligible in Eq. (2). This phenomenon has been found in high moisture foods, fresh fruits, vegetables and insects (Guo et al., 2008; Wang et al., 2005, 2008b).

The slopes of log ε′ vs. log f below 200 MHz in Figs. 3–5 and the electrical conductivity estimated from the intercept of these plots were obtained as a function of temperature for the four compressed legume flour samples at the highest moisture content (19.9–21.6 g/100 g w.b.) used in this study (Fig. 6). The magnitude of negative slopes increased with increasing temperature for all four legume samples. The deepest slopes reached about −0.87 for chickpea and soybean and −0.77 for green pea and lentil (Fig. 6a). This slope was comparable to that for egg yolk (−0.866) over the frequency range from 10 to 1000 MHz with a water content of about 48 g/100 g w.b. (Guo, Trabelsi, et al., 2007). The slope over this frequency range could reach −1 when the moisture content was more than 75 g/100 g in fresh fruits (Wang et al., 2005) and egg white (Guo, Trabelsi, et al., 2007). From Eq. (3), the slope of −1 suggests that the effect of dipole rotation on RF heating was completely negligible. Conversely, the slope became small or close to zero at low temperatures (Fig. 6a),
especially at low moisture contents (Fig. 7) likely due to reduced mobility of charged ions, resulting in low-loss factors (Figs. 3, 4, 5 and 7).

The electrical conductivity estimated from Eq. (4) using dielectric loss factor data in the frequency range of 1–200 MHz at the highest moisture content used in this study is summarized in Fig. 6b. These values increased sharply with increasing temperature for all four legume flour samples. The increasing rate for chickpea was the largest, followed by soybean, lentil and green pea. At 20 °C and below 15.8–17.5 g/100 g moisture content, which corresponded to water activities of 0.70–0.87 (Fig. 2), there was negligible effective electrical conductivity for all four flour samples, suggesting that low temperature and moisture might result in low mobility of charged ions. The corresponding water activity of 0.7–0.8 on the moisture sorption isotherm was close to that for bound water in white breads defined by Liu et al. (2009). Absolute values of effective electrical conductivity for low moisture legume flour samples at 70–90 °C was 2–3 orders of magnitude smaller than that in white breads with the moisture content of 24–38.6 g/100 g at 85 °C (Liu et al., 2009) and 5–6 orders of magnitude smaller than that in high moisture vegetables, fruits and foods at room temperatures (Castro, Teixeira, Salengke, Sastry, & Vicente, 2003; Wang et al., 2005, 2008b).
It is a common believe that an increase in loss factor with increasing temperature in the RF range would result in thermal runaway in RF heating applications. This positive feedback results in higher heating rates for warmer product than for colder product during the RF heating. It is difficult to provide uniform heating when the initial product temperature or the electromagnetic field is not uniform. This could be improved by appropriate designs of RF applicators such as moving product on conveyor belts during RF heating or mixing product between successive RF treatments (Wang et al., 2007a, 2007b).

Dielectric properties of legume samples are much larger than those of air. The compressed legume samples were measured as a solid volume in dielectric measurements. But the voids between legume seeds in actual product volumes may result in nonuniform RF heating. It is reported that the small particle sizes of beans and lentils provides better heating uniformity than walnuts (Wang, Yue, Chen, & Tang, 2008a), which could be helpful in developing potential RF treatments for postharvest insect control in legumes without mixing.

### 3.2. Comparisons of dielectric properties for four legume flours

**Fig. 8** shows a comparison of loss factors among four legume flours for 27 MHz at 20 and 90 °C over four moisture contents. The loss factors of the four legumes increased sharply with increasing moisture contents. At low moisture contents (e.g. <15 g/100 g), the difference in loss factors among four legume samples was small. At 20 °C and the highest moisture contents, the loss factor of soybean was the highest, followed by lentil, green pea, and chickpea (Fig. 8a). However, the loss factor of chickpea at the highest moisture content was very sensitive to temperature, becoming the highest value at 90 °C (Fig. 8b). It will be important to ensure that the moisture content of treated legumes fall within the optimal range for RF heating by pretreatment drying and sorting to improve heating uniformity.

**Fig. 9** shows a comparison of loss factors among four legume flours for 915 MHz at 20 and 90 °C over four moisture contents. Similar to legume flour at 27 MHz (Fig. 8), the loss factors also increased quickly with increasing moisture contents but differences between legumes at both temperatures were smaller. Furthermore, the absolute loss factor values of the four legumes were much smaller than those at 27 MHz.

### 3.3. Density dependent dielectric properties as a function of frequency

The complex-plane plot of chickpea flour samples is shown in **Fig. 10** at 4 commercially important frequencies over all measured temperatures and moisture contents. The slope of the resulting regression lines decreased with increasing frequency and was 0.5508, 0.5021, 0.4725, and 0.4552 for 918, 1206, 1503 and 1800 MHz, respectively. The \( R^2 \) increased with increasing frequency.

<table>
<thead>
<tr>
<th>Legume Flours</th>
<th>Moisture content (g/100 g w.b.)</th>
<th>Penetration depth (cm)</th>
<th>27 MHz</th>
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<th>1800 MHz</th>
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<tr>
<td></td>
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<td>5.4</td>
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</table>
frequency, reaching 0.974, 0.984, 0.990 and 0.995 at 918, 1206, 1503 and 1800 MHz, respectively.

Fig. 11 shows a comparison of complex-plane plots of four legume flour samples at 1800 MHz over all measured temperatures and moisture contents. Differences between the four legume samples were small, especially at low moisture contents and temperatures. The order of slope values was soybean > lentil > chickpea > green pea (Table 2). All four legume samples had a good linear relationship between permittivity and density at 1800 MHz with large coefficients of determination ($R^2 \geq 0.987$). This could be useful for estimating the density of legume samples based on dielectric properties measurements.

3.4. Penetration depth

Penetration depths calculated from the measured dielectric properties of four legume flours are listed in Table 3 at three specific frequencies over three temperatures and four moisture contents. The penetration depth decreased with increasing frequency, temperature and moisture content. The penetration depth in green pea was the least among the four legume samples under the same conditions. Small containers and thin layers of product would have to be used in MW treatments to overcome the lack of penetration at these frequencies. In RF heating, however, legumes could be treated in larger containers and thicker layers due to deeper penetration. Therefore, RF heating may provide practical large-scale and high throughput treatments for postharvest insect control for legumes.

4. Conclusions

Dielectric properties of chickpea, green pea, lentil and soybean flour samples at different frequencies (10–1800 MHz), temperatures (20–90 °C) and moisture contents (about 8–21 g/100 g)/water activities (0.3–0.9) were measured by an open-ended coaxial-line probe and impedance analyzer. The dielectric constant and loss factor of the four legume flours decreased with increasing frequency but increased with increasing temperature and moisture content. At low frequencies and high temperature and moisture contents, negative linear correlations between the loss factor and frequency on a log-log plot suggested that ionic conductance was the dominant heating mechanism. The effective electrical conductivity of legume flour at the highest moisture content and temperature ranged from 1.16 to 2.48 mS/cm, which was 5–6 orders of magnitude smaller than that in high moisture vegetables, fruits and other foods at room temperatures. At high frequencies, all four legumes had a good linear relationship between the permittivity and the density. The difference in loss factors among the four legumes was clear at high moisture contents at 27 MHz and became negligible at 915 MHz for all moisture contents and temperatures. The penetration depth decreased with increasing frequency, temperature and moisture content, which was large enough at 27 MHz to develop large-scale industrial RF treatments.

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