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Temperature and Moisture Dependent Dielectric Properties of Legume Flours Associated with Dielectric Heating

W. Guo (Associate Professor)

College of Mechan. & Electr. Eng., Northwest A&F Univ., Yangling, Shaanxi, 712100, P.R. China

S. Wang (Assistant Research Professor), G. Tiwari (Ph. D Student)

Dept of Management & Operations, Washington State University, Pullman, WA 99164-4736, USA

J. A. Johnson (Research Entomologist)

USDA-ARS-SJVASC, 9611 S. Riverbend Avenue, Parlier, CA 93648, USA

J. Tang (Professor)

Dept. of Biological Systems Eng., Washington State University, Pullman, WA 99164-6120, USA

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Abstract. *Dielectric properties of flour samples from four legumes (chickpea, green pea, lentil, and soybean) at four different moisture contents were measured using an open-ended coaxial probe and impedance analyzer at frequencies of 10 to 1800 MHz and temperatures of 20 to 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 the frequency on a log-log plot, which was mainly caused by the ionic conductance. At 1800 MHz, the dielectric property 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 %). At the highest moisture content (e.g., 20%) soybean had the highest loss factor at 27 MHz and 20°C, followed by green pea, lentil 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.*

Keywords. Dielectric constant; Loss factor; Microwave; Radio frequency; Postharvest

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 productions reached 69,870, 599,416, 147,278, and 87,196,000 metric tons in 2006 with a value of about US\$38, 72, 36, and 18,922 millions, respectively (USDA-FAS, 2007; USDA-NASS, 2007). About 31, 72, 89, and 36% of their total productions are for export to India, Spain, and Latin American countries, such as Colombia, Cuba, and Peru (USDA-FAS, 2007; USADPLC, 2007). A major problem in the 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 disinfestation 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 dry agricultural commodities, such as grain (Nelson, 1984), rice (Lagunas-Solar et al., 2007; Zhao et al., 2007), walnut (Wang et al., 2002, 2007a,b), and wheat (Halverson et al., 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 the product provide general guidance for selecting the optimal frequency range and bed thickness for uniform RF treatments (Wang et al., 2003b). 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 because it has broad frequency bands, is easy to use, and does not need special sample preparations (Sheen and Woodhead, 1999; Wang et al., 2003a). Its well-developed theory makes it possible to obtain sufficiently accurate results both for medium-loss and high-loss materials, such as liquids (Tanaka et al., 2002; Nunes et al., 2006; Guo et al., 2007b), foods (Engelder and Buffler, 1991; Ryyänen, 1995; İçier and Baysal, 2004; Wang et al., 2008c), and fresh fruits (Ikediala et al., 2000; Feng et al., 2002; Wang et al., 2003b, 2005a; Nelson, 2005; Guo et al., 2007a) as affected by temperature and moisture content (Nelson, 1981; Feng et al., 2002; Guo et al., 2008; Wang et al., 2008c). 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 (Nelson, 1984; Nelson and Trabelsi, 2006; Guo et al., 2008). Since dielectric properties of particulate materials depend on sample density (Nelson, 1984; Berbert et al., 2002), the density of the compressed samples should match the true kernel density of the seeds. This method provides relatively accurate measurement of dielectric properties for dry products using open-ended coaxial probe methods (Guo et al., 2008).

Much research has been conducted on dielectric properties of dry products, such as bean (Berbert et al., 2002), coffee (Berbert et al., 2001), flaxseeds (Saciilik et al., 2006), and grain seeds or flours (Nelson et al., 1984; Lawrence et al., 1990; Trabelsi et al., 1998; Nelson and Trabelsi, 2006). 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 flours over the frequency range of 10 to 1800 MHz, temperatures from 20 to 90°C, and 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.

Materials and Methods

Materials and sample preparation

Seeds of green pea, lentil, and soybean were purchased from a local grocery store in Pullman, WA, USA. To accurately determine dielectric properties of the irregularly shaped legume seeds, compressed ground legume flour that matched the true density of seeds was prepared for measurements as described in Guo et al. (2008). Known weights of ground legume flour were placed in a metal cylindrical holder 10 cm in height and 20.7 mm inner diameter (Guo et al., 2008). The flour samples were compressed on a hydraulic presser (Model 3912, Carver, Inc., Wabash, IN, USA) to a desired level (about 3 cm in height) so that the sample density matched the seed density.

Initial moisture contents for the legume flours were 7.9, 10.8, 8.4 and 8.9% (wet basis) for chickpea, green pea, lentil, and soybean, respectively. To obtain samples of four different moisture contents between 7.9 and 21.6%, flour samples were kept over appropriate amounts of distilled water in covered desiccators at room temperature for 3 to 5 days to absorb water. The difference between each moisture content level was about 4%. After achieving the desired moisture contents, each flour sample was mixed, sealed in plastic bags and allowed to equilibrate for two days at room temperature. The moisture content and water activity of each 200 g sample were determined before compressing, and the density of each compressed sample was calculated according to its mass and volume after compressing. The compressed flour samples were kept in plastic bags until used for dielectric property measurements.

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 (C_7H_8) showed little tendency to soak into the sample, flowed smoothly over the sample surface, and has stable gravity and viscosity, it was used as the displacing liquid. The 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 on aluminium moisture dishes in a vacuum oven (ADP-31, Yamato Scientific America Inc., Santa Clara, CA, USA) at 130 °C for one hour according to AOAC (2002). The samples were cooled in a desiccator with $CaSO_4$ at the bottom 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 AquaLab water activity meter (Series 3, Decagon Devices, Inc., Pullman, WA, USA) with an accuracy of $\pm 0.003 a_w$. 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.

Dielectric property measurements

Dielectric properties of the compressed legume flour samples were measured at 200 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 frequency (1800 MHz) is close to another FCC allocated MW frequency (2,450 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% w.b., the dielectric properties were measured at these temperature and moisture ranges. A detailed description of the system can be found in Wang et al. (2003a, b) and Guo et al. (2008). 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 sample 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 because the test cell was water and air tight and the sample temperature was controlled by circulating water (15 L min⁻¹) from a water bath (model 1157, VWR Scientific Products, Niles, IL, USA) into the jacket of the test cell. A 50% water and 50% 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 property measurement, the impedance analyzer was warmed up for at least 1 h and then calibrated with air, a short, a 50Ω load, and a low-loss capacitor. The impedance analyzer and the coaxial probe were further calibrated with a standard air-short-triple deionized water calibration procedure while the cable and probe were maintained at fixed positions during the measurements to minimize errors. Typical error 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 et al., 2003a). Dielectric properties of each sample were measured at 20, 30, 40, 50, 60, 70, 80, and 90°C. 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. Before and after each measurement, the probe and the sample cell were cleaned with deionised water and wiped dry. Dielectric property data for legume flour samples were determined in duplicates. Mean values and standard deviations were calculated from the two replicates.

Effects of sample electrical conductivity and density on dielectric properties

Dielectric loss factors (ϵ'') are mainly dominated by ionic conduction (ϵ''_{σ}) and dipole rotation (ϵ''_d) according to Ryyänen (1995):

$$\epsilon'' = \epsilon''_d + \epsilon''_{\sigma} \quad (1)$$

For liquid or high moisture foods, Eq. (1) becomes:

$$\epsilon'' = \epsilon''_d + \frac{\sigma}{2\pi f \epsilon_0} \quad (2)$$

where f is the frequency (Hz), ϵ_0 is the permittivity of free space or vacuum (8.854×10^{-12} F m⁻¹) and σ is the electrical conductivity (S m⁻¹). If the effect of dipole rotation is negligible at the low frequencies (e.g. ≤ 200 MHz), after setting $\epsilon''_d = 0$ and taking a logarithm on both sides, Eq. (2) becomes:

$$\log \epsilon'' = \log \left(\frac{\sigma}{2\pi \epsilon_0} \right) - \log (f) \quad (3)$$

Eq. (3) shows that a linear relationship between the loss factor and frequency would be established on a log-log plot with a slope of -1. For solid materials of intermediate and low moisture contents, however, the slope was found to be greater than -1 (Wang et al., 2005a; 2008c; Guo et al., 2008; Liu et al., 2009). This suggests that Eq. (3) can not 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 (α). The intercept (α) of the above relationship derived from loss factor data below 200 MHz was used to estimate the effective electrical conductivity of four legume flour samples as a function of temperature and moisture content (Wang et al., 2005a; Liu et al., 2009) as follows:

$$\sigma_e = 2\pi\varepsilon_0 10^\alpha \quad (4)$$

High correlations have been reported between measured dielectric constant ε' and loss factor ε'' divided by the seed density, ρ , for grains and chickpeas in complex-plane plots in frequency range from 1800 to 18000 MHz (Trabelsi et al., 1997; Nelson and Trabelsi, 2006; Guo et al., 2008). This relationship has the following form:

$$\frac{\varepsilon''}{\rho} = a \frac{\varepsilon'}{\rho} - b \quad (5)$$

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 (Trabelsi et al., 1997; Guo et al., 2008). In order to assess the best frequency range for Eq. (5) in this study, the correlation between the measured ε''/ρ and ε'/ρ 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.

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 surface. The penetration depth d_p in m of RF and MW energy in a material can be calculated according to von Hippel (1954). 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.

Results and Discussion

Dielectric properties as influenced by frequency, temperature and moisture

The log-log plot of the dielectric constant and loss factor at different frequencies is presented for compressed flour samples of green pea (Fig. 1) over the temperature range from 20 to 90°C at their highest moisture contents of 21.6%, which was similar for lentil and soybean. 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 (Guan et al., 2004; Nelson, 2005; Wang et al., 2005a, 2008c; Guo et al., 2007a, 2008).

The slopes of $\log \epsilon''$ vs. $\log f$ below 200 MHz in Fig. 1 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 (19.9-21.5%) used in this study (Fig. 2). The negative slopes decreased with increasing temperature for all four legume samples. The lowest slopes reached about -0.87 for chickpea and soybean and -0.77 for green pea and lentil. 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% w.b. (Guo et al., 2007b). The slope over this frequency range could reach -1 when the moisture content was more than 75% in fresh fruits (Wang et al., 2005a) and egg white (Guo et al., 2007b). 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. 2a), especially at low moisture contents because the charged ions were less mobile in the RF range, resulting in low loss factors.

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 is summarized in Fig. 2b. 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% moisture content, which corresponded to water activities of 0.70-0.87, 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 34-38.6% at 85°C (Liu et al., 2009) but 5-6 orders of magnitude smaller than that in high moisture vegetables, fruits and foods at room temperatures (Castro et al., 2003; Guan et al., 2004; Wang et al., 2005a; 2008c).

The increase in loss factor with increasing temperature in the RF range would result in thermal run away in RF heating applications. This positive feedback

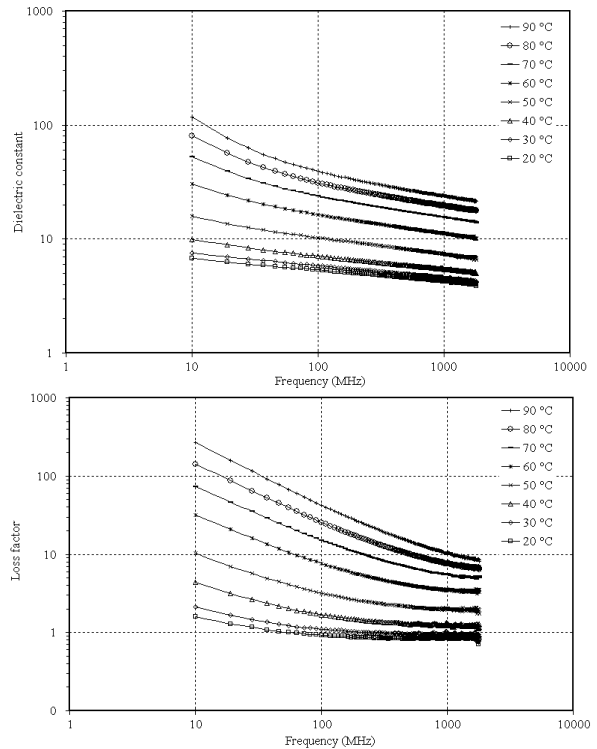


Fig. 1. Frequency dependence of the dielectric properties of compressed green pea flour samples over the temperature range from 20 to 90°C at the highest moisture content of 21.6%

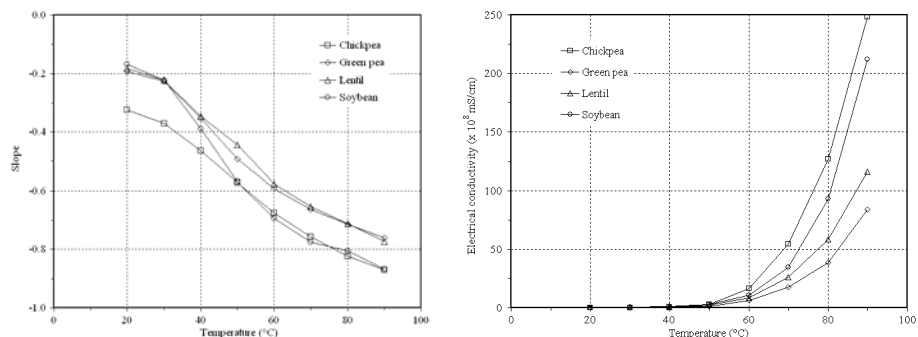


Fig. 2. Slope (a) of $\log \epsilon''$ vs. $\log f$ (<200 MHz) in Figs. 3-5 and electrical conductivity (b) as a function of temperature of four compressed legume flour samples at the highest moisture content

results in higher heating rates for warmer product than for colder product during the RF heating (Wang et al., 2006). 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 (Wang et al., 2008a) such as moving product on conveyor belts during RF heating or mixing product between successive RF treatments (Wang et al., 2005b; 2007a, b).

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 non-uniform RF heating. It is reported that the small particle sizes of beans and lentils provides better heating uniformity than walnuts (Wang et al., 2008b), which could be helpful in developing potential RF treatments for postharvest insect control in legumes without mixing.

Density dependant dielectric properties as a function of frequency

The complex-plane plot of chickpea flour samples was conducted 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, reaching 0.974, 0.984, 0.990 and 0.995 at 918, 1206, 1503 and 1800 MHz, respectively. Complex-plane plots of four legume flour samples were compared 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 1). 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 property measurements.

Table 1. Coefficients of the linear relationship (Eq. 5) between permittivity and sample density of four legume samples at 1800 MHz

Legumes	<i>a</i>	<i>b</i>	R^2
Chickpea	0.4552	0.8046	0.995
Green pea	0.4410	0.6241	0.996
Lentil	0.4609	0.6347	0.991
Soybean	0.5048	0.8869	0.987

Penetration depth

Penetration depths calculated from the measured dielectric properties of four legume flours are listed in Table 2 at three specific frequencies over three temperatures and four moistures 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, it could be expected that 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.

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%)/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 the frequency on a log-log plot suggested that ionic conductance was more important than dipole

rotation. The effective electrical conductivity of legume flours at the highest moisture content and temperature ranged from $1.16\text{-}2.48 \times 10^{-6}$ mS cm⁻¹, 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 penetration depth decreased with increasing frequency, temperature and moisture content, which was large enough at 27 MHz to develop large-scale industrial RF treatments.

Table 2. Penetration depths (cm) calculated from the measured dielectric properties of four legume flours at three specific frequencies over three temperatures and four moistures contents

Legume flours	Moisture (% w.b.)	Penetration depths (cm)								
		27 MHz			915 MHz			1800 MHz		
		20°C	60°C	90°C	20°C	60°C	90°C	20°C	60°C	90°C
Chickpea	7.9	1792.9	1073.1	136.2	54.2	32.0	8.6	22.2	12.4	4.3
	11.4	1595.1	902.0	74.1	38.7	21.1	6.6	19.4	8.8	3.4
	15.8	948.1	116.5	17.3	25.4	8.1	2.7	15.5	3.9	1.6
	20.9	449.3	32.7	8.8	17.5	4.6	1.9	9.1	2.6	1.3
Green pea	10.8	1176.8	388.7	37.3	27.1	11.7	3.9	14.5	4.8	2.0
	14.6	947.6	205.0	23.9	21.6	9.5	3.1	10.9	4.3	1.7
	17.5	697.5	88.0	18.6	16.1	6.2	2.6	7.4	2.9	1.5
	21.6	361.5	52.3	14.6	13.0	4.9	2.4	6.5	2.4	1.5
Lentil	8.4	1850.2	1039.3	91.5	47.4	26.0	6.7	27.8	12.4	3.5
	12.9	1097.1	278.7	31.1	27.3	11.2	3.4	9.4	4.6	1.9
	16.9	730.2	123.7	18.9	16.6	7.2	2.4	7.8	3.2	1.4
	21.5	431.6	42.4	12.1	16.5	4.5	2.0	7.3	2.4	1.3
Soybean	8.9	1989.6	564.0	83.3	35.2	17.5	6.8	26.5	6.8	3.2
	11.7	1280.3	203.5	27.9	35.1	12.6	4.1	12.4	5.3	2.4
	16.0	593.0	83.7	16.6	21.5	7.6	2.7	9.3	3.6	1.7
	19.9	270.5	39.9	9.6	14.3	5.4	1.9	6.7	2.8	1.3

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