Dielectric properties of papaya seeds from 75 kHz to 5 MHz**

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Received July 20, 2018; accepted December 18, 2018

Abstract. The object of this work was to study the dielectric properties of papaya seeds of the Golden cultivar and seeds of two papaya hybrids, Tainung and Calimosa, in the frequency range from 75 kHz to 5 MHz at four levels of bulk density, and with moisture contents ranging from 6 to 23% wet basis. The relative permittivity and the loss factor of the seeds were measured using a precision LCR meter, and their relationship with the variable frequency of the oscillating electric field, the seed moisture content and bulk density, and the cultivar/hybrid type were established. Relative permittivity for each value of the moisture content was reduced regularly as the frequency increased. Abrupt changes in the slopes of the curves demonstrating the relationship between the relative permittivity and the loss factor, and the moisture content were considered an indication of changes in the water sorption mechanisms occurring within the seed. The relationship between the relative permittivity and the loss factor, and bulk density was represented by linear functions with positive slopes. The effect of the cultivar or hybrid types on the relative permittivity and the loss factor revealed that seeds of the Golden cultivar and the Tainung hybrid exhibited similar relative permittivity values in the whole frequency range studied, in contrast to seeds of the Calimosa hybrid.

Keywords: Carica papaya L., dielectric loss factor, moisture content, relative permittivity, seed bulk density

INTRODUCTION

The papaya tree (Carica papaya L.) is a typically tropical fruit tree with a probable centre of origin between the southern region of Mexico and Costa Rica (Kim et al., 2002). However, this tree has become widely distributed in several areas of the world, with reports of its production in virtually all tropical countries and warm climate subtropical regions (Fuentes and Santamaria, 2014). The fruit is considered relatively exotic in temperate countries, whereas papaya is an important vitamin source in countries with tropical climate. In addition, proteinases extracted from the latex of green fruits are widely used in the pharmaceutical and food industries (Carvalho and Renner, 2012).

The annual global production of papaya is greater than 12 million t, with a continuous growth rate of 367 000 t per year in the last 15 years (FAO, 2017). In Brazil alone, with annual production of 1.6 million t (FAO, 2017), the estimated use of papaya seeds is approximately 5 000 kg per year with a market value of US$ 2 200 kg⁻¹ for the Calimosa hybrid papaya seeds and US$ 3 500 kg⁻¹ for the Tainung hybrid papaya seeds (Carlesso, 2009). The values mentioned above demonstrate the importance of an in-depth study of the steps involved in the processing of papaya seeds, including their handling, drying and storage, all of which involve the accurate determination of the moisture content of the seeds. Thus, it is pertinent to include the study of the dielectric properties of papaya seeds in the research aimed at determining the on-line moisture content of agricultural commodities of greater commercial expression, such as cereal grains, oilseeds and coffee.

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Although papaya seeds exhibit variable physiological behaviours in relation to storage conditions (Sun and Liang, 2001), papaya seeds were initially classified as orthodox by Ellis et al. (1985). However, this classification was re-evaluated by Ellis et al. (1991) when stating that the behaviour of papaya seeds in storage should be redefined as an intermediate between those observed in orthodox and recalcitrant seeds. Orthodox and intermediate seeds are generally stored for extended periods of time with reduced moisture contents (5 to 8%) (values of the percentage moisture content are expressed on a wet basis (w.b.) throughout the paper) at low temperatures.

Moura et al. (2013) emphasized the importance of the on-line moisture content sensors installed in the storage environment that would function as a method of quality control of the product in storage, without the need to destroy the samples, as is the case with the oven drying method. The on-line moisture content meters of granular agricultural materials are classified as indirect measurement methods because they are based on physical properties that can be correlated with the volumetric concentration of water in the seed sample. Currently, meters that use dielectric properties as parameters for estimating the moisture content are the most widely studied. According to Trabelsi et al. (2013), it is necessary to analyse in detail the relative permittivity ($\varepsilon'$) and the loss factor ($\varepsilon''$) as a function of frequency, along with bulk density ($\rho$) and the temperature of seed samples contained inside the capacitive sensors to correlate dielectric properties with the seed moisture content. This procedure predicts which frequencies and ranges of the moisture content and bulk density may integrate dielectric models that shall be incorporated into electronic measurement systems. Therefore, in order to determine the moisture content with accuracy, the effects of both bulk density and temperature must be made explicit through additional measurements and compensation, or eliminated by the identification of functions that are independent of bulk density and insensitive to temperature variations (Trabelsi et al., 2013).

Most researchers looking for solutions to reduce the effect of bulk density on the on-line determination of the moisture content of agricultural products have concentrated their efforts on the development of meters operating at microwave frequencies (Digman et al., 2012; Lisovsky, 2007). This focus is partly due to the decrease or elimination of problems related to ionic conductivity, which allows the study of a wider range of moisture contents. However, researchers have also demonstrated that it is possible to indirectly measure the seed moisture content (generally up to 23%), regardless of the bulk density variations of the sample, using radiofrequencies (Funk et al., 2007; Moura et al., 2013). This is due to the fact that, with the appropriate choice of the size and shape of electrodes, it is possible to measure the moisture content using a considerable volume of seeds, resulting in a more representative measurement.

To design a capacitive sensor that can integrate an online moisture content meter for agricultural purposes, and that is as comprehensive as possible, it is first necessary to determine the dielectric properties of various types of products. The investigation of the variation of $\varepsilon'$ and $\varepsilon''$ as a function of the moisture content (M), along with bulk density ($\rho$) and the electric field oscillation frequency (F) makes it possible to determine the ranges or values of these variables, which will compose the dielectric model $M = f(\varepsilon', \varepsilon'', \rho, F)$ that is independent of the variation of $\rho$. In fact, studies on the dielectric properties of seeds, such as rice (Prasad and Singh, 2007), maize (Csársenyo et al., 2013; Sacilik and Colak, 2010), sorghum (Moura et al., 2013) and wheat (Berbert and Stenning, 1996); oilseeds, such as peanuts (Boldor et al., 2004), flax seed (Sacilik et al., 2006) and soybean (Trabelsi and Nelson, 2006); legumes, such as beans (Berbert et al., 2002); coffee (Berbert et al., 2001); and wheat flour mixed with oat meal (Luczycka et al., 2013) have been performed over the last decades, using both microwave frequencies and radiofrequencies.

Thus, when designing a capacitive sensor that can integrate the electronic circuit of an on-line moisture content meter prototype of granular agricultural products, there should be new dielectric functions that would allow the estimation of the moisture content of as many products as possible, to make it more comprehensive. The inclusion of new dielectric functions is also useful for calibrating the meter as new varieties or cultivars enter the market (Trabelsi and Nelson, 2006).

Considering the above, the objective of the present study was to determine the dielectric properties of papaya seeds of the Golden cultivar, and the Tainung and Calimosa hybrids for the moisture content values ranging from 6 to 23% at four levels of bulk density, for frequencies between 75 kHz and 5 MHz, using automatic data collection methods through software specifically developed for this purpose. In this study, the effect of temperature on dielectric properties was not evaluated, constituting the subject to be studied in a forthcoming paper on dielectric models for moisture estimation.

**MATERIAL AND METHODS**

The papaya fruits (Carica papaya L.) of the Solo group, cv. Golden, from the F2 generation of the Calimosa hybrid and the Tainung hybrid were harvested at maturation stage 2 in a commercial crop of the company CalimanAgricola S/A located in the municipality of Linhares, State of Espírito Santo, Brazil (19°23’28"S latitude, 40°04’20"W longitude, altitude: 33 m). This stage of maturation is characterised by the fact that the fruit peel surface has 15 to 25% of a yellowish colour (CEAGESP, 2015). Fruits are harvested in early stages of maturation to reduce the loss of firmness during the subsequent handling and transportation. Besides, papaya harvested at maturation stage 2 had
received higher scores in sensory evaluation, mainly flavour and appearance, by the time it reached market shelves, which generally coincides with full ripeness (Bron and Jacomino, 2006). We collected 45 fruits of each genotype. Subsequently, the fruits were transported to the Laboratory of Agricultural Engineering of the Centre for Agricultural Sciences and Technologies of the State University of Northern Rio de Janeiro, located in the municipality of Campos dos Goytacazes, State of Rio de Janeiro, Brazil (21°45'15"S latitude, 41°19'28"W longitude, altitude: 13 m). When they arrived at the laboratory, the fruits were placed on a bench and remained at ambient conditions for approximately 10 days until reaching maturation stage 5, which is the stage in which the fruits are fully ripe, with more than 75% of the yellowish peel surface (CEAGESP, 2015). Seeds obtained from fruits at maturation stage 5 have higher germination and vigour, as compared to seeds from unripe fruits (Martins et al., 2006).

The fruits were cut longitudinally to remove the seeds and placental remnants, which were then transferred to a steel mesh sieve. Cleaning was performed by friction against the mesh of the sieve in running water until the placenta and sarcotesta were completely removed. The seeds were exposed to laboratory ambient conditions for approximately 3 h over a wire sieve until the surface water evaporated. The pre-drying procedure reduced the moisture content of the seeds to approximately 40%. Subsequently, the seeds were dried at 40°C in a prototype fixed-bed dryer with tangential air flow until the moisture content was reduced to approximately 23%. The seed lot was then divided into sub-lots that were packed in sealed 1750 ml glass containers and stored in a BOD incubator at 10°C.

To obtain sub-lots with the desired moisture content values, namely between 23 and 6%, in each experiment, one of the stored containers was removed from the refrigerated environment and remained on the bench until it was in thermal equilibrium with the ambient air. Afterwards, the seeds were arranged on four rectangular trays with perforated bottoms and sides to form a thin layer with a height corresponding to the average thickness of the seeds. Then, the trays were placed inside the prototype dryer, where they were maintained for the time necessary to reach the expected moisture content. The monitoring of the drying process and, therefore, the moisture content was performed gravimetrically by weighing the tray every 5 min, using a digital scale with 0.01 g accuracy. After obtaining the desired moisture content, the seeds were again stored in the glass container at 10°C for 10 days to homogenise the moisture content. The seed moisture content was determined using the oven-drying method at 103°C for 17 h (ISTA, 2010).

Because there are no rules or established specifications for evaluating the physiological quality of papaya seeds, procedures proposed in the Rules for Seed Analysis (Brasil, 2009) were adapted for this study. The germination test was performed using four replicates of 50 seeds each. The seed moisture content prior to the germination test was 40%. Seeds were placed in germination test paper rolls moistened with a quantity of water equivalent to 2.5 times the paper mass, and placed in a germinator for an 8 h light/16 h dark daily photoperiod, at 30°C/20°C, respectively, with the photon flux density of approx. 2.5 W m⁻² from warm white fluorescent tubes. Evaluations were performed on day 7 (the first count) and day 28 (the final count), and the results were expressed as the percentage of germinated seeds. In this way, the physiological quality (the first and final counts, respectively) of the seeds used in this experiment was as follows: the Golden cultivar (33.5 and 34.5%), the Tainung hybrid (69.5 and 71.5%) and the Calimosa hybrid (31.0 and 36.5%).

A Hewlett-Packard 4285A precision LCR meter was used for the measurement of electrical parameters, capacitance and conductance. This equipment is capable of measuring 12 impedance parameters at test frequencies, from 75 kHz to 30 MHz. The dielectric properties were determined using an effective voltage of 1.0 Vₑₑ. A four-terminal pair configuration was used to connect the concentric cylindrical capacitor to the HP 4285A meter. This type of connection minimises problems related to mutual inductance, contact resistances and electrical noise.

The sensor used to hold the samples during measurements of the dielectric properties of the seeds was a concentric cylindrical capacitor, similar to that described by Berbert et al. (2002). The working volume of the capacitor is 5.5 x 10⁻⁴ m³, and it is able to hold approximately 0.09 kg of papaya seeds with 13% moisture content.

The two complex electrical permittivity parameters were calculated by measuring the equivalent capacitance in parallel (Cₑₑ), and the conductance (G) of both the empty sensor and the sensor filled with papaya seeds. The Cₑₑ and G values were measured at 75, 80, 85, 90, 95, 100, 200, 300, 400 and 500 kHz, and 1, 2, 3, 4 and 5 MHz. The ε’ and ε” values were calculated according to the methodology described by Moura et al. (2013). Automatic data collection was performed using a computer with an HP-IB interface card and LabVIEW (National Instruments) control software. In this study, procedures similar to those reported by Berbert and Stenning (1996) were adopted to determine the effect of the bulk density of the granular material on its dielectric properties. For each moisture content level, samples with four bulk density values were obtained. The mean values of temperature and relative humidity in the laboratory during the experiment were 23.3±1.4°C and 59.7±7.8%, respectively.

RESULTS AND DISCUSSION

Figure 1 present the variation curves of ε’ and ε”, respectively, of the seeds of the Tainung hybrid as a function of frequency for the moisture contents in the range of 9.4 to 23.1%, and bulk density of 145±1 kg m⁻³ at 23±1°C and
56 ± 3% relative humidity. The variation pattern of $\varepsilon'$ and $\varepsilon''$ as a function of the electric field oscillation frequency was similar for the other genotypes. Thus, a reduction of $\varepsilon'$ and $\varepsilon''$ occurred with an increase in the frequency from 75 kHz to 5 MHz. These results were similar to those reported for corn (Sacilik and Colak, 2010) and buckwheat (Zhu et al., 2013). We also verified that $\varepsilon'$ was higher for the samples containing higher moisture contents for the same frequency. In comparison, in the samples of the Tainung hybrid with the moisture contents of 9.4% (145 kg m$^{-3}$) and 23.1% (147 kg m$^{-3}$), at 100 kHz, the value of $\varepsilon'$ increased from 1.46 to 3.88, which represents an increase of 166%. The samples with moisture contents lower than 12% displayed lower variations in the values of $\varepsilon'$ and $\varepsilon''$ as a function of frequency.

The fact that an increase in the frequency caused a decrease in both the $\varepsilon'$ and $\varepsilon''$ values (Fig. 1) can be explained by noting that when a dielectric material is subject to polarization by an alternating current electric field, for each direction reversal of the field force lines, the dipoles try to realign themselves due to this change of direction in a process that requires some finite time. For each type of polarization, there is a minimum reorientation time that depends on the ease with which the specific dipoles are able to realign. The relaxation frequency is calculated as the inverse of this minimum reorientation time. A dipole cannot maintain the change in the direction of its orientation when the frequency of the applied electric field exceeds its frequency of relaxation; therefore, it will not contribute to relative permittivity $\varepsilon'$. In addition, the energy losses related to the value of $\varepsilon''$ are also reduced (Blythe and Bloor, 2005). The water molecules present in the papaya seeds are of a dipole nature; thus, they have an asymmetric charge centre. The dipoles are constantly influenced by the polarity change of the electric field, although they are typically randomly oriented. Thus, the electric field tries to drag and align them. However, when the electric field ceases to act, the dipoles return to their initial condition (Kraus and Carver, 1984; Schiffmann, 2014).

The behaviour of the dielectric loss factor as a function of frequency (Fig. 1B) was similar though less regular than that observed for the $\varepsilon'$ values, mainly for higher values of the moisture content. Similarly, in general, a decrease in the $\varepsilon''$ value as a function of an increase in the electric field oscillation frequency was noted for the same value of the moisture content. For the same frequency, the $\varepsilon''$ values were increased for wetter samples. The relationship between $\varepsilon''$ and frequency became more complex for moisture contents greater than 14.8% (Calimosa and Tainung) and 16.2% (Golden) (Fig. 1B). In these cases, there was a sudden change in the $\varepsilon''$ curve for frequencies near 200 kHz. The dispersion of the $\varepsilon''$ values as a function of frequency was more pronounced for higher values of the moisture content and lower frequency values, a behaviour pattern also observed for flaxseeds (Sacilik et al., 2006) and safflower seed (Sacilik et al., 2007).

Figure 2 represent the variations of $\varepsilon'$ and $\varepsilon''$, respectively, as a function of the moisture content of the Golden cultivar seed samples (164±2 kg m$^{-3}$) for four representative frequency levels. Similar curves were obtained for seeds of the Calimosa and Tainung hybrids (145±1 kg m$^{-3}$). For the same frequency value, $\varepsilon'$ increased with the increasing moisture content, and this increase was more intense for higher values of the moisture content and lower frequency values, a behaviour pattern also observed for flaxseeds (Sacilik et al., 2006) and safflower seed (Sacilik et al., 2007).
As expected, $\varepsilon'$ became less sensitive to the variation in the moisture content when the frequency approached 5 MHz. In most cases, $\varepsilon'$ and $\varepsilon''$ are virtually independent or vary insignificantly as a function of the frequency and the moisture content of up to 12-13%. However, as the moisture increases, there is a sharp change in the slope of the curves showing the variation of $\varepsilon'$ and $\varepsilon''$ as a function of the moisture content. Noticeable changes in the slopes of the $\varepsilon'$ and $\varepsilon''$ vs. moisture curves for common bean (Berbert et al., 2002) and parchment coffee (Berbert et al., 2001) have occurred at somewhat higher values of moisture, i.e., from 16 to 18%, and from 15 to 16%, respectively. The changes in permittivity and the loss factor, as a result of changes in moisture, are generally considered an indication of changes in the binding forces that exist between a monolayer of water molecules bound to the surface of the cells that form the walls of the capillaries within the seed (strongly bound water), and between adsorbed water molecules and molecules of water vapour, i.e., less tightly bound water or free water molecules (Kraszewski, 1996).

If the previous assumption is correct, the change in the slope of the equilibrium moisture content curve for papaya seeds should occur at a moisture content value close to the values indicated by the dielectric dispersion, i.e., 12-13%. Indeed, by experimentally determining the 30°C isotherm for papaya seeds of the Tainung hybrid from 0 to 90% relative humidity (rh), the change in the slope occurred at 10% moisture, and the equilibrium relative humidity was 70%. For the lower portion of the isotherm, namely 40 to 55% rh, the regression of the equilibrium moisture content ($M_{eq}$) on rh yielded a straight line ($M_{eq} = 0.0516 \text{rh} + 5.109$) with a coefficient of determination of 0.9818. The corresponding results for the interval $70\% \leq \text{rh} \leq 90\%$ were $M_{eq} = 0.1922 \text{rh} – 3.416$, with a coefficient of determination of 0.9846.

Variations in measurements of the permittivity and the loss factor of papaya seeds of the Calimosa hybrid at 1.0 MHz, at different levels of bulk density, are presented in Fig. 3. Measurements were obtained in a laboratory at $23\pm1^\circ\text{C}$ and $58\pm7\%$ relative humidity. The relationships between $\varepsilon'$ and $\varepsilon''$, and $\rho$ for the seeds of the Golden cultivar and the Calimosa hybrid at 5.0 MHz were obtained by the least squares method, and were found to be linear. For these two genotypes, an increase in the moisture content resulted in an increase in the slope of the lines representing the relationship of $\varepsilon'$ and $\varepsilon''$, and bulk density. This behaviour indicates that the influence of $\rho$ on the studied
dielectric properties is intensified by an increase in the moisture content of the samples. However, for the seeds of the Tainung hybrid with high moisture content, the relationship between $\varepsilon'$ and $\varepsilon''$, and bulk density was best described by second-degree polynomial functions.

Considering that, theoretically speaking, the line representing the relationship between $\varepsilon'$ and $\rho$ converges to a point whose Cartesian co-ordinates are very close to (0,1), linear regression was performed to relate the relative permittivity of the seeds of the three genotypes to bulk density values at 1.0 MHz (at 5 MHz for seeds of the Golden cultivar) for the moisture content values ranging from 6.6 to 23.1%. Thus, when the sample container is empty and $\rho$ has a value of 0 kg m$^{-3}$, the permittivity approaches a mean value of 0.9842±0.0176. This value is very close to unity, which is the established value of the permittivity of air (Kraus and Carver, 1984). The coefficients of determination of the linear equations relating permittivity and bulk density for the mentioned moisture range varied from 0.8919 to 0.9998. Again, a family of straight lines relating $\varepsilon''$ and $\rho$ should theoretically converge to a point whose Cartesian coordinates are very close to (0,0), i.e., when the sample container is empty, the loss factor approaches a value very close to zero, given that air is considered a lossless substance. Indeed, for moisture contents in the range from 6.6 to 23.1%, the straight lines relating the loss factor and bulk density for the three genotypes at 1.0 MHz (at 5 MHz for seeds of the Golden cultivar) converges to 0.0058±0.0061 with coefficients of determination varying in the range 0.7622 ≤ $r^2$ ≤ 0.9985.

Papaya genotypes also had some effect on the dielectric properties, and its influence upon $\varepsilon'$ and $\varepsilon''$ is presented in Fig. 4, for samples at 18.8±0.2% moisture and an average bulk density of 145.2±1.2 kg m$^{-3}$. Tests were conducted under laboratory conditions at 25.2±0.6°C and 65.3±7.9% relative humidity. As noted in Fig. 4, the highest values of permittivity and the loss factor occurred for the Calimosa seeds, whereas the lowest corresponding values were obtained for the Tainung and Golden seeds in approximately the entire range of the frequencies studied. The shapes of the curves representing the variation of the loss factor on frequency were not as regular as those representing the variation of permittivity on frequency for the three genotypes. Permittivity varied from 2.22 (mean; Golden and Tainung) to 2.51 (Calimosa), whereas the loss factor varied from 0.3909 (mean) to 0.5082 at 1.0 MHz. These variations correspond to changes of 13% in permittivity and 30% in the loss factor.

Although seeds of papaya genotypes may differ in their chemical analysis, it is unlikely that a slight change in the chemical composition within different genotypes may significantly affect the dielectric properties, given that most organic compounds are nonpolar. The effect of the papaya genotype on the $\varepsilon'$ and $\varepsilon''$ values may be accounted for by differences in the physical properties of the seeds associated with each genotype. According to Kupfer (1996), at higher frequencies, the size of the seeds within bulk samples of granular materials influences the absorption and scattering of the electromagnetic field, thus affecting their dielectric properties. In the present work, for similar values of bulk density, the measured values of capacitance and conductance increased as a result of larger grain dimensions, thus increasing the $\varepsilon'$ and $\varepsilon''$ values. Indeed, considering that its shape can be approximated by a prolate spheroid, i.e., the shape that is formed when an ellipse rotates around its major axis, the largest seed dimensions (the major axis × the minor axis) occurred for the Calimosa hybrid (5.26 × 3.71 mm). In contrast, the lowest corresponding values were obtained for the Golden cultivar (4.81 × 3.50 mm) and the Tainung hybrid (4.87 × 3.50 mm). Samples of 50 seeds of each genotype were used to calculate the mean seed dimensions.

Due to the effect of the genotype on the dielectric properties, it is likely that in the possible use of $\varepsilon'$ and $\varepsilon''$ in the estimation of the moisture content of papaya seeds, using the capacitive method, different calibration curves may be needed, depending on the type of the genotype used.

![Fig. 4](image-url) Fig. 4. Effect of the genotype on permittivity $\varepsilon'$ (A) and loss factor $\varepsilon''$ (B) of papaya seeds at a 18.8±0.2% moisture, 25±1°C, and a relative humidity of 65±8%, and the indicated values of bulk density. ○ – Calimosa (145.2±1.9 kg m$^{-3}$); X – Golden (146.4±2.0 kg m$^{-3}$); □ – Tainung (144.1±1.5 kg m$^{-3}$).
CONCLUSIONS

1. The relative permittivity and dielectric loss factor values for each of the moisture content values of papaya seeds, and for similar bulk density values of the samples, regularly decrease with the frequency increasing from 75 kHZ to 5 MHz.

2. The relationship between the loss factor and the field oscillation frequency becomes more complex for moisture contents greater than 17-19%, depending on the genotype. In these cases, a sudden change is noted in the variation curve of the loss factor for frequencies below 200-300 kHz.

3. The relative permittivity and the dielectric loss factor are virtually independent of the frequency and the moisture content for values up to 14-15%. However, as the moisture increases, a sharp change is noted in the slope of the curve that depicts the variation of the relative permittivity and the loss factor as a function of the moisture content. This change is considered an indication of the alteration in the water adsorption mechanisms inside the seed.

4. The relationship between the relative permittivity and the loss factor, and bulk density is generally represented by increasing linear functions. However, the slopes of the lines representing these functions increase as the moisture increases. This behaviour demonstrates that the influence of the bulk density on the studied dielectric properties is intensified by an increase in the moisture content of the samples.

5. The influence of the genotypes on the dielectric properties indicates that in the possible use of the relative permittivity and the loss factor to estimate the moisture content of papaya seeds, using the capacitive radio-frequency method, different calibration curves are necessary as a function of the genotype used.

ACKNOWLEDGEMENTS

Thanks are due to Caliman Agricola S.A. which provided all the fruit lots used in this experiment. We would like to acknowledge the contribution of Mr Pedro Henrique Dias for the collection of fruits.

Conflict of interest: The authors declare that they have no conflict of interest.

Compliance with Ethical Requirements: This research does not contain any experiment involving human or animal subjects.

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P.A. BERBERT et al.


