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Dielectric properties of egg components and microwave heating for in-shell pasteurization of eggs

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Abstract

In this study, microwave heating has been considered for in-shell egg pasteurization. In the first part, the effects of temperature (0–62 °C) and frequency (200 MHz to 10 GHz) on the dielectric properties of egg components were investigated. In the second part, individual egg components as well as intact in-shell eggs were brought to pasteurization temperature in a laboratory-scale microwave oven working at 2450 MHz using different (0.75, 1 and 2 W g^{-1}) power densities and the heating curve was analyzed to determine heating time required for different power levels. Under the conditions studied, it was demonstrated that the albumen had higher dielectric properties and loss factors leading to its faster heating rate in a microwave environment than the yolk. This was corroborated by the microwave heating trials performed on individual components where albumen always heated up faster. Laboratory trials on microwave heating of in-shell eggs indicated that, on the contrary, the heating rates of both albumen and yolk were similar. Microwave heating appeared to have great potential for in-shell egg pasteurization. Models for calculating the ε' and ε'' at a given frequency and temperature for shell egg components were also presented.

Keywords: Pasteurization; Shell eggs; Microwave; Heating patterns; Dielectric properties

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1. Introduction

Eggs are potential hosts and carriers for pathogenic microbes like *Salmonella enteritidis,* due to their rich nutritive value. Heat pasteurization is considered the best solution for controlling these pathogens (FSIS-USDA, 2006). Egg is an excellent food supplement, giving almost every essential amino acid which most of our regular diet may lack. It is also an excellent source of vitamin A, B_3 and Folate. It also contains useful amounts of many other vitamins and minerals (Li-Chan, Powrie, & Nakai, 1995). Egg is used as a vital ingredient in several foods and food industries, especially for their exceptional functional properties. These properties mainly depend on the protein quality of the eggs and are severely affected when heated, due to protein denaturation. Thus a heat pasteurization technique with minimal changes to these proteins needs consideration.

Eggs are one among the major animal foods mostly marketed raw and frequently consumed raw. More than 90% of food borne Salmonellosis, caused by *Salmonella enteritidis* is through shell eggs (Schroeder et al., 2005). Most of the *Salmonella enteritidis* outbreaks generally involved grade A eggs that are washed and disinfected and also met the requirements of the state for shell quality (St. Louis, Morse, & Potter,. 1988).

The Food Safety and Inspection Service (FSIS) of United States Department of Agriculture (USDA) recommends heating the egg white and the egg yolk to 57.5 °C and 61.1 °C, respectively, for 2.5 min to ensure egg safety against *Salmonella* and other food borne pathogens (FSIS-USDA, 2006). This is possible by conventional heating methods only if the yolk and egg white are separated before processing. But breaking and repacking them aseptically involves huge additional costs. Therefore in-shell

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pasteurization has gained great commercial importance in recent times.

The current technique for in-shell pasteurization of egg involves heating the eggs in a water bath at 60 °C for about 20–25 min, depending on the size of the eggs. This leads to the overheating of the egg white proteins (i.e., the egg white gets heated up more than the yolk, which is against the recommendations) resulting in denaturation and coagulation (Hou, Singh, Muriana, & Stadelman, 1996). This denaturation greatly affects the functional properties of the eggs (Van der Plancken, 2005). Therefore a process that can heat the shell eggs from inside will be a better alternative to solve this problem.

Microwave heating exploits the dielectric behaviour of the substance exposed to it to generate heat from within the substance. But this direct heat generation occurs only up to a certain depth of the product from the surface. This is due to the fact that depending on the dielectric properties of the substance, there is an exponential decay of microwave energy as the waves penetrate into the product from the surface (Meda, Orsat, & Raghavan, 2005). The yolk contains the main reserve of food substances, required for the development of embryo. The albumen is the chief reservoir of water and it is the most alkaline of all the natural liquids (Lokhande, Arbad, Landge, & Mehrotra, 1996). Theoretical mathematical studies have shown that even though albumen exhibits better dielectric properties than yolk, the egg's curvature has a focusing effect which leads to a suitable power distribution (Datta, Sumnu, & Raghavan, 2005). The shell egg appears ideally suited for pasteurization in a microwave environment (Fleischman, 2004; Rehkopf, 2005).

Measurements of dielectric properties (dielectric constant and dielectric loss factor) are required for understanding, explaining, and empirically relating physico–chemical properties of the material microwave energy (Venkatesh & Raghavan, 2005). Dielectric properties vary with the composition, moisture content and temperature of the food and the frequency of the electric field. Information on the dielectric properties of albumin and yolk is limited. Modelling changes in dielectric properties of the albumen and yolk with temperature and frequency will allow predicting the same at any prescribed temperature and frequency thereby facilitating equipment design and process optimization to ensure best end product quality. A complete understanding of the dielectric properties and egg curvature on power distribution will help design a system highly specific and efficient for this application (Liao, Raghavan, Dai, & Yaylayan, 2003).

Therefore this study was conducted with the following objectives:

(i) To measure the dielectric properties of albumen and yolk of eggs in the frequency range of 200 MHz to 10 GHz and in the temperature range of 0 °C to pasteurization temperatures (57.5 °C for egg white and 61.1 °C for yolk).

(ii) To study heating rates and time taken to reach the above mentioned pasteurization temperatures from ambient temperature (come-up time) for a given power level in the laboratory microwave oven at 2450 MHz for albumen and yolk, in and out of the egg-shell, ignoring the non uniformity factor in microwave heating and to verify the shell integrity while heating to pasteurization temperatures.

2. Materials and methods

In this study, the potential benefits of using microwave energy for heating fresh in-shell eggs to pasteurization temperatures were investigated. At first, dielectric properties of egg albumen and yolk were measured at temperatures ranging from 0 °C to the required pasteurization temperature, and at frequencies ranging from 200 MHz to 10 GHz. Empirical relationships were then obtained to express dielectric properties as a function of temperature and frequency. In the second part, egg white and yolk as well as intact in-shell eggs were heated from 24 °C to the temperature required for pasteurization in a laboratory-scale microwave oven. Effects of power levels on heating rates and egg quality were measured and compared.

2.1. Measurement of dielectric properties of the egg components

2.1.1. Egg samples

The fresh whole eggs, within three days of grading and packing (identified from the best before date stamped on the eggs), used in this study were procured from the local market and kept in a refrigerator set at 5 °C until used. They were all of Canadian grade A and of large size with an average weight of 60 g each. These eggs were marketed from an egg grading station that uses chlorinated water at ambient temperature to wash the eggs before grading.

2.1.2. Equipment

Measurements of the dielectric properties were made with the open ended coaxial probe technique (Agilent 8722 ES *s*-parameter Network Analyzer equipped with a high temperature probe model 85070B, Santa Clara, USA) (Venkatesh & Raghavan, 2005) and controlled by a computer software (Agilent 85070D Dielectric Probe Kit Software Version E01.02, Santa Clara, USA). According to the manufacturer, the equipment has an accuracy of ±5% for the dielectric constant (ε') and ±0.005 for the loss factor (ε'') (HP, 1992). A diagram of the experimental setup used for the measurement of dielectric properties is shown in Fig. 1.

The dielectric constant and loss factor were calculated from the load admittance $Y_{\mathrm{L}}(\omega, \varepsilon)$ directly by the software. The expression used by the software for calculating $Y_{\mathrm{L}}(\omega, \varepsilon)$ is as follows (Venkatesh & Raghavan, 2005):

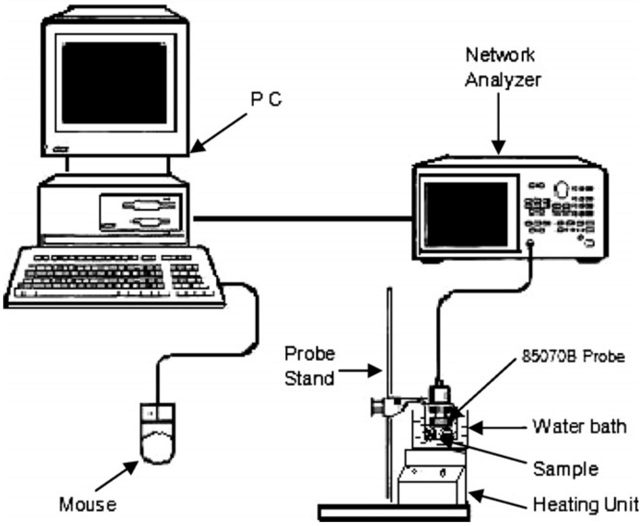

Fig. 1. Experimental setup for the measurement of dielectric properties.

$$Y_{\mathrm{L}}(\omega,\varepsilon) = Y_0 \frac{1-\Gamma(\omega,\varepsilon)}{1+\Gamma(\omega,\varepsilon)} \tag{1}$$

where $Y_{\mathrm{L}}(\omega,\varepsilon)$ is load admittance in S; ω is the operating angular frequency in Hz; ε is the overall permittivity in F/m; Y_0 is the characteristic admittance of probe of 50 Ω; and $\Gamma(\omega,\varepsilon)$ is the measured reflection coefficient.

2.1.3. Experimental design and procedure

Prior to measurement, the eggs were cracked carefully and the egg white (35 g) and yolk (20 g) were collected separately in small cylindrical beakers. Egg white was homogenised by slow stirring with a glass rod and deal with a single entity for the measurements and not as thin and thick albumen. All measurements were made in triplicates. Egg white from an individual egg formed one replicate and egg yolk from an individual egg formed one replicate, whereas the shell and shell membrane from two eggs formed one replicate in order to make up the minimum volume required for taking measurements.

The shell and shell membrane were separated by soaking in water at ambient temperature for 1 h. Moisture content was determined by oven method using representative samples before and after the soaking operation with a regular hot air oven set at 105 °C (Thermo Electron Corp #6528, Marietta, USA). The samples were dried to the initial moisture content ($\pm 1\%_{\mathrm{w.b}}$) using the same oven before taking the dielectric property measurements, thus making sure that there was no significant increase in the moisture content, affecting the measured values of the dielectric properties. The shell was finely powdered using a mortar and pestle to an average particle size less than 250 μm (USA standard test sieve E-11 specification No. 60). The membrane was carefully folded to the required thickness of measurement (6 mm). All the prepared samples were refrigerated to 0 °C in the freezer of a domestic refrigerator (Kenmore, Chicago, USA) by continuously monitoring with the help of an electronic thermometer (Maverick #RF-02, Edison, USA) (±0.1 °C precision) and used immediately.

The samples to be measured were taken in small cylindrical beakers (20 mm in diameter, 50 mm in height and 2 mm thick borosilicate glass). The probe was mounted on the stand, facing downwards. Calibration of the probe was done using a shorting block, air and water before each experiment and then checked by measuring distilled water to ensure the calibration was stable.

The samples were heated using a water bath (custom made with an electric hot plate (Fisher Scientific, USA) and a 500 ml glass beaker (Borosil, Waukegan, USA)) set at desired temperatures (i.e., 60 °C for egg white, 65 °C for egg yolk, shell and shell membrane to compensate for the losses and to obtain the required pasteurization temperatures in the samples) and placed right beneath the probe on the platform of the stand used to mount the probe (Fig. 1). Measurements were taken every 5 °C interval (chosen tentatively as there were no anomalies identified between consecutive measurements) from 0 to 57.5 °C for egg white (i.e., 5 °C interval till 55 °C and at 57.5 °C), 61 °C for yolk and 60 °C for the shell and shell membrane. The temperature of the samples was measured using an alcohol thermometer (±0.1 °C precision) attached parallel to the probe and held at a distance of 2 mm from the probe on the same stand with its bulb completely immersed in the sample. The samples were stirred well with a glass rod before every measurement to ensure uniformity. As the changes in the dielectric properties are heating time dependant, the measurements were taken with continuous heating at the rate of $2\,^{\circ}\mathrm{C\,min^{-1}}$ with less than 5 s intermittence only during measurements to ensure there is no temperature change during the measurements (3 s each). Any irreversible changes that occurred during the heating process were ignored.

The dielectric properties were measured at 100 different frequencies ranging from 200 MHz to 10 GHz.

2.1.4. Data analysis

MATLAB version 7.01 was used to analyze the collected data and to establish the mathematical relationships for the dielectric constant and loss factor as a function of frequency and temperature.

2.2. Comparing microwave heating rates of egg white and yolk in and out of the shell

2.2.1. Egg samples

Commercially available large size fresh eggs were procured from the local market in refrigerated conditions and allowed to stay at the room temperature for about 3–4 h to reach the ambient temperature of about 24 °C. These eggs were marketed from an egg grading station that uses chlorinated water at ambient temperature to wash the eggs before grading. Samples of egg white and yolk were collected in a similar fashion as for the measurement of dielectric properties. The shell and shell membrane were discarded. For in-shell heating trials shell eggs weighing 60 ± 1 g were used.

2.2.2. Equipment

An instrumented and computer controlled laboratory-scale microwave (MW) oven (custom built in the laboratory) (Fig. 2) was used for this part of the study. Its main components were: a 2450 MHz microwave generator (Gold Star 2M214, South Korea) with adjustable power from 0 to 750 W, waveguides, a three-port circulator, a manual three-stub tuner to match the load impedance, microwave couplers to measure forward and reflected power, a carbon load to absorb reflected power and a microwave cavity made of brass, (47 × 47 × 27 cm) in which the egg samples were processed. The wave guides were rectangular (72 × 35 mm) and TE_{10} mode of application was used.

The microwave generator (magnetron) produced microwaves with varying power densities based on the supplied power. The generated microwaves were guided using the waveguides into the microwave cavity via the above mentioned components in a sequence. The manual three-stub tuner was used to adjust the reflected power, thereby keeping it at the minimum possible value (<10% of the incident power). The temperatures were measured using fibre optic probes (Nortech EMI-TS series, Quebec City, Canada). The probes were connected to a data acquisition unit (Agilent 34970A, Santa Clara, USA) which was again connected to a computer. The entire setup was monitored and controlled using the HPVEE (Agilent, Santa Clara, USA) object oriented programming language.

2.2.3. Experimental design and procedure

All measurements were taken in triplicates (each replicate obtained from an individual egg, except for the yolk wherein the yolk from two eggs were combined for each replicate). The samples of egg white and yolk (40 g each) were placed (one at a time) inside the microwave chamber in small (50 ml) cylindrical glass beakers (20 mm in diameter, 50 mm in height and 2 mm thick borosilicate glass) and heated to the pasteurization temperatures of 57.5 °C and 61.1 °C, respectively.

Also, probes were introduced through the shell of the in-shell eggs (one for the white and one for the yolk) tentatively, assuming that the yolk was located approximately at the center of the in-shell egg and egg white along the sides surrounding the yolk and heated in the microwave chamber with the broad end of the egg facing upwards, till the yolk reached 61.1 °C with the same experimental design. The precise locations of probes were later confirmed by boiling the egg with the probes in it, followed by peeling and slicing the boiled egg and carefully examining the location of the probes. Any measurements taken from eggs that had some discrepancy in the probe location were discarded. In both the above mentioned cases, temperature measurements were recorded at an interval of 5 s each (but temperature was continuously monitored every second by the computer for precise heating control) and the experiment was repeated for different power densities (0.75, 1 and 2 W g^{-1}). A single point measurement is considered representative of the temperatures due to very low power densities used and slow heating rates obtained. Also any non-uniformity in heating is not investigated for. The egg white, yolk and shell egg were held at the pasteurization temperatures (±0.5 °C) for 2.5 min with repeated microwave heating cycles controlled by the computer running HPVEE object oriented programming language.

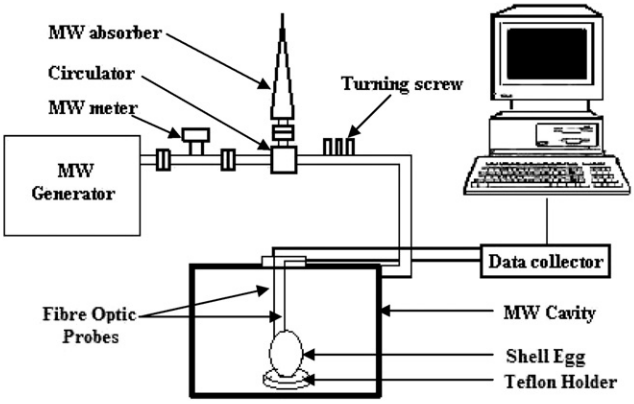

Fig. 2. Experimental setup for microwave heating.

Hot spots and cold spots which are characteristic of microwave heating were ignored in placing the probes, as the standard deviation of the heating time for consecutive measurements was small.

2.2.4. Post-treatment shell integrity (visual observation)

Tests were also conducted without any probe and the eggs were examined for any visible cracks or deformations in the egg-shell, following the same experimental design and procedure mentioned above. The time of microwave application was determined by the heating rate obtained from the previous trials with the probe. A scale ranging from 0 to 5, of which 0 being no cracks and 5 being a broken/leaking egg was used to measure the visual shell integrity of the egg.

2.2.5. Data analysis

Temperature measurements obtained for the egg white and yolk for a given power level were plotted as a function of temperature versus time to observe the heating curves within the pasteurization temperatures to study the actual heating time required for in-shell eggs to reach pasteurization temperatures and the heating rates for different power densities (0.75, 1 and 2 W g^{-1}) were compared. The data obtained were used to calculate the come-up time (the time taken to reach the pasteurization temperatures) for egg white, yolk and the shell egg at different power densities mentioned above.

3. Results and discussion

3.1. Dielectric properties of the egg constituents

Both ε' and ε'' for egg white and yolk appeared linearly related to temperature and frequency in a similar fashion as that of water and also the dielectric properties of the egg white appeared to be much closer to that of water (Collie, Hasted, & Ritson, 1948).

As the egg white has nearly 90% water (Li-Chan et al., 1995), the ε' and ε'' values obtained for egg white were much closer to that of water than those observed for the yolk. At any given temperature and frequency, repeatability of the measurements was excellent and the variances calculated among replicates were smaller than 0.15.

The ε' for both egg white and yolk decreased with increasing temperature and frequency (Eqs. (3) and (5)) whereas the ε'' decreased with increase in temperature and increases with increase in frequency (Eqs. (4) and (6)). A linear additive model was used to relate ε' or ε'' to temperature and frequency. Its general form of the relationship was

$$(\varepsilon' \text{ or } \varepsilon'') = a + b \cdot T \pm c \cdot F \tag{2}$$

where T is the temperature in °C, F is the frequency in GHz, and a, b, c are the model coefficients.

Regression analysis performed on the collected data yielded the following relationships for egg white:

$$\varepsilon' = 72.38 - 0.17 \cdot T - 1.75 \cdot F \quad (R^2 = 0.925) \quad (P < 0.01) \tag{3}$$

$$\varepsilon'' = 17.22 - 0.19 \cdot T + 1.58 \cdot F \quad (R^2 = 0.948) \quad (P < 0.01) \tag{4}$$

and for egg yolk

$$\varepsilon' = 50.085 - 0.13 \cdot T - 1.72 \cdot F \quad (R^2 = 0.925) \quad (P < 0.01) \tag{5}$$

$$\varepsilon'' = 13.55 - 0.11 \cdot T + 0.65 \cdot F \quad (R^2 = 0.905) \quad (P < 0.01) \tag{6}$$

Values of the regression coefficient (R^2) in Eqs. (3)–(6), which are close to 1, indicate that the models have excellent predictability and the probability of getting a different value is less than 1% *($p < 0.01$)*. These models are useful in determining the dielectric properties of egg yolk or egg white at any given temperature and frequency within the range studied. Graphical representations of the measured values of ε' and ε'' for the egg white and yolk can be found in Figs. 3 and 4. The above models were evaluated for their accuracy and adaptability with another set of measurements taken and found to have a perfect fit.

The yolk samples had lower values than the egg white for both ε' and ε''. This was attributed to the fact that the yolk contains lower moisture of around 47 $\%_{\text{w.b}}$ (Li-Chan et al., 1995) when compared to the egg white. Both ε' and ε'' of the yolk were less sensitive to temperature and frequency than the egg white (Fig. 4). The values obtained were comparable to those obtained by Ragni et al. for the eggs within the three days of storage (Ragni, Al-Shami, Mikhaylenko, & Tang, 2007). Fig. 5 shows the variation of ε' and ε'' with temperature for 2450 MHz frequency.

The ε' and ε'' values of both the shell and shell membrane did not change significantly with respect to temperature and frequency. The ε' and ε'' values remained relatively constant around 3.5 and 0.5, respectively. As a result, both the shell and the shell membrane are relatively transparent and permeable to microwaves. This was attributed to the low moisture content of both shell and shell membrane which was only $9\%_{\text{w.b}}$ and to a lesser extent the composition and structure of the shell proteins; and to the fibre matrix of shell and shell membrane might be responsible for this good transparency to microwaves (Calvery, 1933; Sasikumar & Vijayaraghavan, 2006).

The penetration depth (D_p) of the microwave power (P) for attenuation by a factor of $1/e$ for is given by (Meda et al., 2005)

$$D_p \approx \frac{\lambda_0 \sqrt{\varepsilon'}}{2\pi\varepsilon''} \tag{7}$$

where λ_0 is the wavelength of the microwave used = 122.2 mm for 2450 MHz

By applying the values of the ε' and ε'' obtained above, the depth over which the electric field (E) falls by a factor of $1/e$ (i.e.) skin depth ($d = 2D_p$) for the egg white is approximately 19.4 mm and for the yolk is approximately 21.3 mm.

3.2. Microwave heating of individual egg constituents

Egg white and yolk samples were first heated separately in the laboratory microwave oven. Because of their higher dielectric property values, egg white samples heated up faster than the yolk samples (Fig. 6). The egg white took about 4.1 (±0.24), 6.9 (±0.4) and 9 (±0.35) min with average heating rates of 14.02, 8.33 and 6.39 °C/min for the microwave power densities of 2.0, 1.0 and 0.75 W g^{-1}, respectively, whereas the yolk required about 9.1 (±0.32), 14.1 (±0.64) and 16.9 (±0.48) min with average heating rates of 6.71, 4.33 and 3.61 °C/min, respectively, for the same microwave power densities mentioned above.

Heating rates for the power density of 2 W g^{-1} were significantly different ($p < 0.05$) from that of both 1 W g^{-1} and 0.75 W g^{-1}, except that the pasteurization temperatures were attained 2–3 min faster using 1 W g^{-1} than 0.75 W g^{-1}. The heating was more even, without any coagulated protein lumps when heated at low power (0.75 or 1.0 W g^{-1}) of microwave. At higher power densities, heating was uneven and lumps of coagulated egg white were observed. This showed that heating of shell eggs to temperatures higher.

3.3. Microwave heating of in-shell egg

There was no significant difference ($p < 0.05$) in the come-up time (time taken to reach the pasteurization temperature) for the egg white heated in beaker and in-shell, although it exhibited a significantly different ($p < 0.05$) heating curve with different power densities. However, the yolk had heated up faster in-shell than in a beaker. It was further noted that at any power density studied, the time to reach pasteurization temperatures were similar

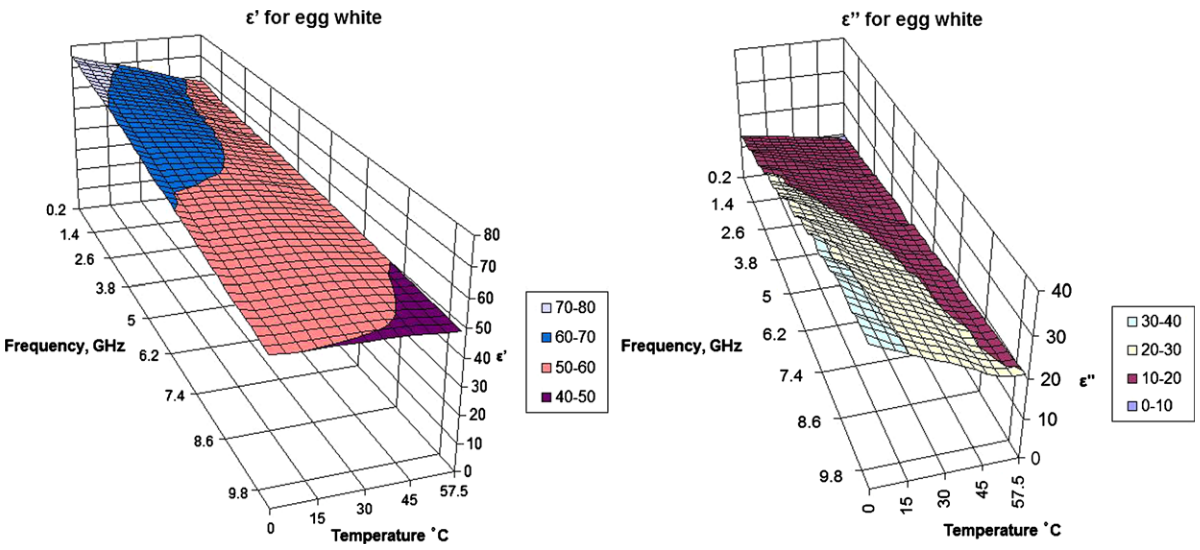

Fig. 3. ε' and ε'' values for egg white.

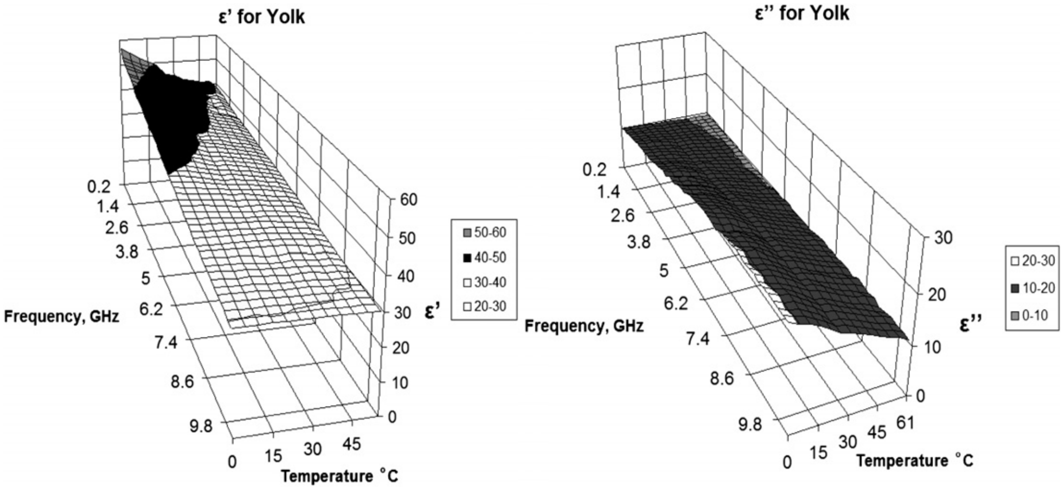

Fig. 4. ε' and ε'' values for egg yolk.

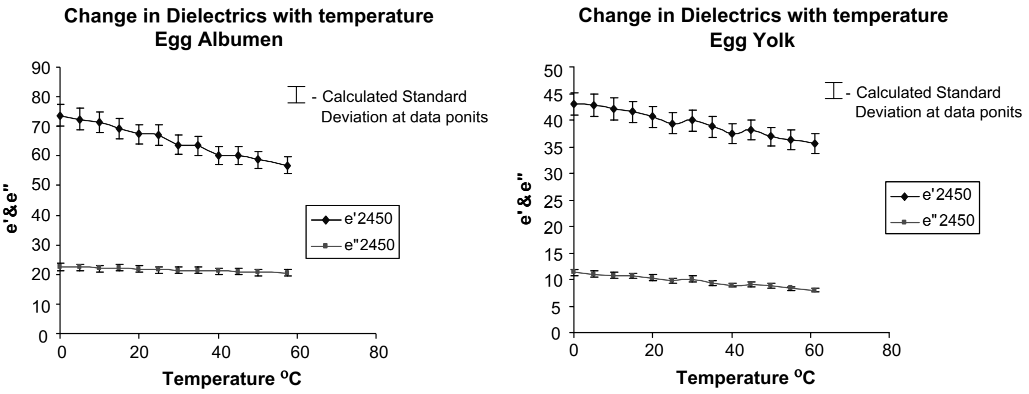

Fig. 5. Change in dielectric properties with temperature for 2450 MHz.

for both the yolk and the albumen indicating that microwave heating is definitively suited for in-shell egg pasteurization. The overall time to reach the pasteurization temperatures during in-shell heating was found to be about 3.5 (±0.26), 7 (±0.46) and 9 (±0.52) min for power densities of 2.0, 1.0 and 0.75 W g^{-1}, respectively, (Fig. 7).

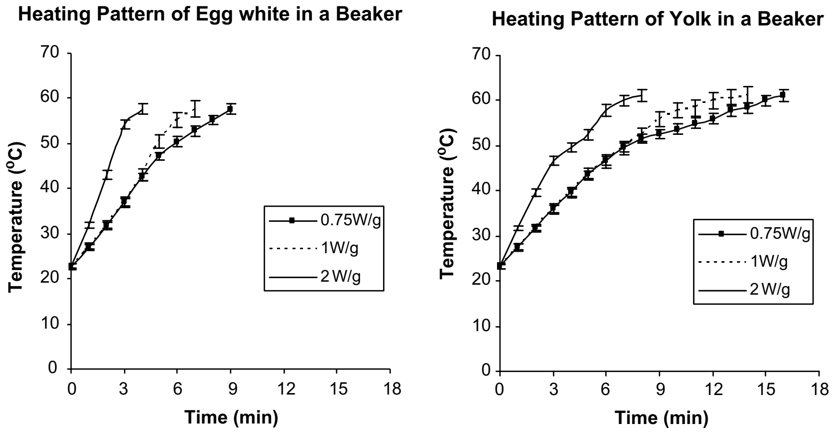

Fig. 6. Heating curves of egg white and yolk in a beaker at different microwave power levels.

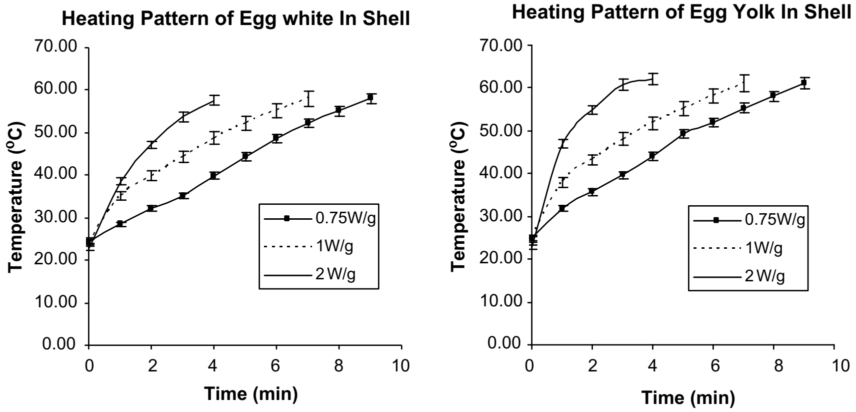

Fig. 7. Heating curves of egg white and yolk in in-shell eggs at different microwave power levels.

Possible reasons for this phenomenon have been proposed by Datta et al. (2005). They suggested that the focusing effect of the egg-shell curvature, the spherical geometry and the central yolk position inside a shell egg have resulted in a convergence of the microwave energy towards the center hence increasing heat dissipation in the yolk (Datta et al., 2005). In addition, the radial penetration depth and loss/attenuation of the microwave energy could have contributed to the higher heating rate of the yolk. The visual examination showed no crack or any deformation on the shell surface.

The higher yolk heating rates were also observed at higher power density (Fig. 7). But higher power densities also led to greater non-uniformity in heat/energy distribution within the shell resulting in localized overheating. This was evident from the number of small coagulated lumps increasingly found in both egg white and yolk with increasing power densities. This also showed that heating of shell eggs to temperatures higher than the pasteurization temperatures (represented by the coagulated lumps, apparently due to overheating) is possible without losing shell integrity.

3.3.1. Post-treatment shell integrity (visual observation)

All the intact shell eggs (without any probes) heated for the same duration for different power densities as observed in the previous trials with probes, remained absolutely intact without any cracks. Thus all the eggs scored ‘0’ without any deviation in the visual test for shell integrity.

4. Conclusions

The study of dielectric properties has provided a good qualitative understanding of the behaviour of the egg constituents under microwave heating. A regression model was developed to assist in further investigation and development of a microwave in-shell egg pasteurization unit. The overall dielectric behaviour of the major egg components (egg white and yolk) were found similar to that of the trend followed by water for the frequency and temperature limits of this study. The egg-shell and shell membrane showed very good transparency to microwaves in their dielectric properties, thereby making the egg-shell a suitable container for microwave pasteurization.

Also the heating curves of the egg components in and out of the shell gave a lucid picture of the heating behaviour of shell eggs in a microwave environment and a clear idea of the heating time required for different power levels. It was interesting to note that the yolk, which had poorer dielectric properties, heated up a little faster than the egg white when heated within the shell. Combination of egg geometry, dielectric properties, and size could be the main factors responsible for the enhanced interior heating. But this requires further investigation. This information can be useful in further investigations and in designing appropriate equipment to accomplish the task of microwave pasteurization of shell eggs. The issues of non-uniformity and the complexity in scaling up these findings to industrials scales requires more detailed investigation into various other aspects. Microwaves appeared to do better in terms of the process time and product quality when compared to the current water bath pasteurization technique. Microwaves have the potential for in-shell egg pasteurization.

Acknowledgements

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