International Journal of Food Properties, 16:917–927, 2013
ISSN: 1094-2912 print / 1532-2386 online
DOI: 10.1080/10942912.2011.567428

EFFECTS OF ACID, SALT, AND SOAKING TIME ON THE DIELECTRIC PROPERTIES OF ACIDIFIED VEGETABLES

Craig B. Koskiniemi[1], Van-Den Truong[2], Roger F. McFeeters[2], and Josip Simunovic[1]

[1]*Department of Food, Bioprocessing and Nutrition Sciences, North Carolina State University, Raleigh, North Carolina, USA*

[2]*USDA-ARS, SAA Food Science Research Unit, Department of Food, Bioprocessing and Nutrition Sciences, North Carolina State University, Raleigh, North Carolina, USA*

In order to design a continuous microwave process for pasteurization of acidified vegetables, equilibration phenomena in acid and salt solutions must be examined with regards to changes in dielectric properties. The objective of this study was to examine the effects of acid and salt concentration on the dielectric properties of acidified vegetables. Broccoli florets and sweet potato cubes (1.2 cm) were blanched to facilitate acid and salt equilibration by heating for 15 s in boiling de-ionized water. Red bell pepper cubes were not blanched. The vegetable samples were then acidified in solutions of 1–2% sodium chloride with 0.5–2% citric acid. Dielectric properties were measured at 915 MHz from 25 to 100°C after 0-, 4-, and 24-h soaking periods in the solutions using an open-ended coaxial probe connected to a network analyzer. Equilibration occurred within 4 h of salting and acidification. Acid and salt concentration had no significant effect on the dielectric constant (ε'). However, ε' was significantly different among vegetables ($p < 0.05$). Dielectric loss factor (ε'') was not affected by acid, but significantly increased with salt concentration. These results provide necessary dielectric property information to apply microwave heating technology in processing of acidified vegetables.

Keywords: ***Dielectric properties, Acidified vegetables, Sweet potatoes, Red bell pepper, Broccoli, Microwave heating.***

INTRODUCTION

The use of microwave processing technology for pasteurization of acidified vegetables presents potential advantages over conventional thermal processing techniques. The application of microwave energy provides rapid heating of both liquid and particulate phases based on dielectric properties, as opposed to conventional methods, which rely on heat transfer by convection and conduction. As a result, microwave energy can potentially shorten heating times, minimize over-processing, and reduce water and energy usage.[1] Therefore, microwave processing technology is well-suited to thermal processing

Received 23 August 2010; accepted 27 February 2011.

Address correspondence to Van-Den Truong, USDA-ARS, SAA Food Science Research Unit, Department of Food, Bioprocessing and Nutrition Sciences, North Carolina State University, Raleigh, NC 27695-7624, USA. E-mail: den.truong@ars.usda.gov

of acidified vegetables. However, one of the first steps in determining the feasibility of microwave processing for any food involves the determination of dielectric properties.

Dielectric properties provide insight with regards to the absorption of electromagnetic energy and its conversion into heat. Collectively, the term dielectric properties refers to the dielectric constant (ε') and dielectric loss factor (ε''). The dielectric constant is a real quantity indicative of the capacity of a material to store electromagnetic energy. The dielectric loss factor is an imaginary quantity, and indicates the ability of a material to convert the absorbed electromagnetic energy into heat. Both ε' and ε'' of a food may change as a function of temperature, frequency, moisture content, salt/ash content, density, structure, and other compositional differences.[2]

Oftentimes foods undergo treatments prior to thermal processing. Thus, it is important to understand the influences of these treatments on the dielectric properties of food materials. In the case of acidified vegetables, such pretreatments include blanching and equilibration of the product in a solution containing acid and salt. Within these treatments, factors, such as acid and salt concentrations as well as the equilibration time, may affect dielectric properties, and in turn influence microwave heating. The influence of salt on the dielectric properties of vegetables and numerous other foods has been well documented, and is known to increase the dielectric loss factor.[3] With regards to blanching and equilibration treatments, Sarang et al.[4] were able to reduce electrical conductivity variation, thereby improving heating uniformity of a chicken chow mein through selective blanching treatments of food components in a highly conductive, salt-containing sauce prior to ohmic heating. This finding by Sarang et al. is highly relevant to dielectric heating since electrical conductivity is a major component of the dielectric loss factor.

While much information exists on the influence of salt on dielectric properties of vegetables, there is no literature available on the dielectric properties of acidified vegetables. With regards to acidulants used in food systems, two studies have examined the dielectric properties of different vinegars[5,6] and reported detectable differences in dielectric spectra as a function of acetic acid concentration. On the other hand, Lau and Tang[7] used 915 MHz microwaves in a batch process to pasteurize pickled asparagus with improved textural properties. However, no information on dielectric properties was presented and, therefore, a full understanding of the role of dielectric properties in liquid-solid mixtures could not be gained. The objectives of this study were to determine the effects of salt and acid concentrations, and soaking time on the dielectric properties of broccoli, red bell pepper, and sweet potato.

MATERIALS AND METHODS

Vegetable Preparation

Broccoli, red bell pepper, and orange-fleshed sweet potato (cultivar *Covington*) were used in this study. Broccoli florets were prepared by cutting into pieces of 3 ± 1 cm in length, and 2 ± 1 cm in width. Red bell peppers were cored and sweet potatoes were peeled prior to dicing into 1.2-cm cubes using the 3/8 in. slicer plate and the 3/8 in. dicer plate of a Hobart food processor (Model FP150, Hobart, Troy, OH, USA).

Titration of Vegetable Materials

Titration of vegetable tissues with citric acid was necessary in order to determine the quantity of acid required to achieve an equilibrated pH of 3.8. Vegetable tissue and de-ionized water (1:1 w/w ratio) were blended in a Waring blender (Model 700S, Waring Product Corp., New York, NY, USA) for 2 min then titrated with 2.70 M of citric acid.

Blanching

In order to facilitate the acidification of the vegetables to pH 3.8 within 24 h, blanching was evaluated. Broccoli, red bell pepper, and sweet potato were cut and diced and separated into two groups: unblanched and blanched. Blanching involved submersion of the vegetables in 90°C water for 15 s as determined in preliminary experiments, and then cooling in an ice bath for 2 min. The vegetable pieces were submerged in citric acid and sodium chloride (NaCl) solutions at a 50:50 w/w ratio for red bell pepper and sweet potato and 30:70 w/w ratio for broccoli. Broccoli required a greater amount of cover solution due to the size and structure of the broccoli florets, which prevented the broccoli pieces from being packed together closely. The pH of vegetable samples was measured after 4 and 24 h equilibration periods. Results from these preliminary experiments indicated that blanching of broccoli and sweet potato was required to reduce pH to 3.8 within 24 h. Blanching was not required for red bell pepper. Therefore, based on the rate of acidification, the present study focused on the dielectric measurements of blanched broccoli and sweet potato, and unblanched red bell pepper.

Equilibration

Food grade citric acid (Thermo Fisher Scientific, Inc., Waltham, MA, USA) and NaCl (Sigma Aldrich, St. Louis, MO, USA) were used in the cover solutions to acidify and salt the vegetables. Cover solutions were prepared by weighing NaCl and citric acid monohydrate into a volumetric flask, and diluting to volume with deionized water. The concentrations of NaCl and citric acid used are shown in Fig. 1. Sweet potato and red bell pepper (500 g) and broccoli (300 g) were placed in separate glass jars. A cover solution

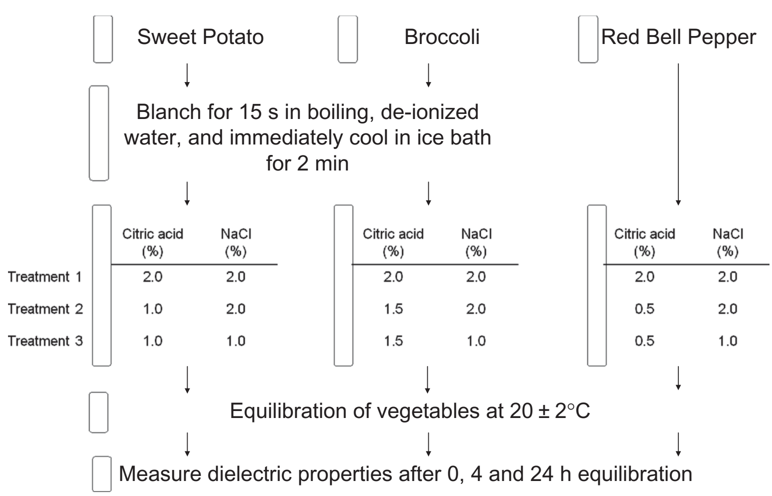

Figure 1 Diagram of experimental procedure. Vegetable:solution ratios are 50:50 for sweet potato and red bell pepper, 30:70 for broccoli.

(500 g) was then added to the sweet potato and red bell pepper to achieve the 50:50 ratio of vegetables to cover solution, and 700 g of cover solution was added to the broccoli to achieve a 30:70 ratio. Jars were then capped and allowed to equilibrate at 20 ± 2°C.

Measurements of Dielectric Properties

Two 25-g vegetable samples were drawn after 0, 4, and 24 h of holding in the cover solution at 20°C for dielectric property measurements. The treatment summary for determining the effects of salt and acid concentrations and experimental design are illustrated in Fig. 1. Samples were mashed and placed into a test cell. Dielectric constants and loss factors were measured at 915 MHz using a network analyzer (HP 8753C, Agilent Technologies, Palo Alto, CA, USA) with an open-ended coaxial probe (HP 85070B, Agilent Technologies) in a pressurized test-cell to prevent loss of moisture. The test cell was submerged in an oil bath (Model RTE111, Neslab Instruments Inc., Newington, NH, USA) and the dielectric properties of the samples were measured in 15°C intervals from 25–100°C. The temperature of the oil bath was gradually increased such that the temperature difference between the sample and the oil bath did not exceed 20°C. Dielectric property measurements were replicated for each treatment combination. Each measurement took less than 10 s.

Statistical Analysis

Statistical analysis was performed using the mixed procedure (PROC MIXED) in SAS version 9.1 (SAS Institute Inc., Cary, NC, USA). Type three analysis of variance was used to determine differences in ε' and ε'' with respect to vegetable type, salt and acid levels, temperature, soaking time, and their interactions.

RESULTS AND DISCUSSION

Dielectric Properties of Raw Vegetables

The dielectric properties of raw broccoli, red bell pepper, and sweet potato were measured to establish an initial time point prior to acidification. The dielectric constants (ε') and loss factors (ε'') as a function of temperature are shown in Fig. 2. Dielectric constants for red bell pepper were the highest, followed by sweet potato and broccoli. Conversely, red bell pepper had the lowest values of ε'', followed by broccoli and sweet potato. Statistical analysis (Table 1) showed that ε' and ε'' were significantly different between vegetables over the temperature range from 25 to 100°C. For each vegetable, ε' decreased and ε'' increased with increasing temperature. This means that as the temperature rose, the ability of the vegetables to store electromagnetic energy decreased, but its ability to convert electromagnetic energy into heat increased. These findings were consistent with previous reports,[8,9] which showed the same trends in ε' and ε'' for vegetables at 915 MHz as temperature increased. Interestingly, ε'' of sweet potato was markedly greater than both red bell pepper and broccoli. The greater values of ε'' for sweet potato were likely a result of the higher sugar content[9] or other chemical constituents, such as organic acids and minerals contributing to the ionic conductivity that is commonly referred as ionic loss, since the moisture content of sweet potato was lower (79.5% f.w.b.), compared to 88.4 and 92.8% moisture for broccoli and red bell pepper, respectively.

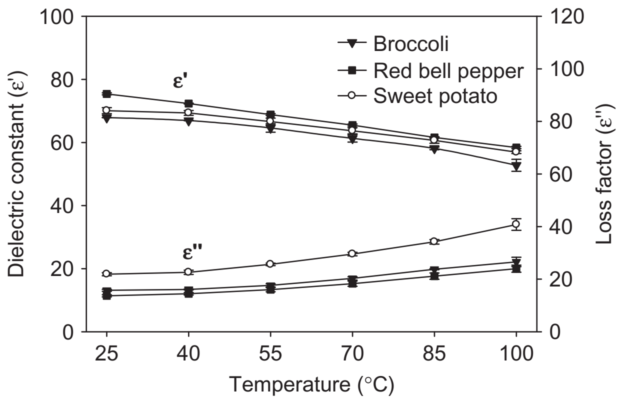

Figure 2 Dielectric constants (ε') and loss factors (ε'') at 915 MHz as a function of temperature for broccoli, red bell pepper, and sweet potato. Error bars represent one standard deviation ($n = 2$) and are too small to be seen.

Table 1 Analysis of variance for ε' and ε''.

Effect[a]	Dielectric constant, ε', Pr > F	Loss factor, ε'', Pr > F
Veg	0.0020	<0.0001
Acid (Veg)	0.3772	0.0615
Salt (Veg)	0.9049	< 0.0001
Time	0.2653	<0.0001
Temp	<0.0001	<0.0001
Time*Temp	0.0365	<0.0001
Veg*Time	0.5120	0.0615
Time*Acid (Veg)	0.8079	0.1928
Time*Salt (Veg)	0.9934	0.0019
Temp*Acid (Veg)	0.5197	0.0248
Temp*Salt (Veg)	0.5493	<0.0001
Time*Temp*Salt (Veg)	0.9866	0.0008
Time*Temp*Acid (Veg)	0.4549	0.0448

[a]Veg = vegetable type; Temp = temperature.

Effect of Soaking Time on Dielectric Properties

The diffusion of intracellular solutes into the cover solution and migration of salt and citric acid into the vegetable tissue is a dynamic, time-dependent process. Preliminary experiments showed that the pH of unblanched tissues of broccoli and sweet potato could not be lowered to 3.8 within 24 h when equilibrated in a cover solution at room temperature. Therefore, a blanching step was implemented to disrupt the cellular structure and facilitate the movement of salt and citric acid into the vegetable tissue to more rapidly lower the pH. The blanching procedure was used for broccoli and sweet potato; however, no blanching step was used for red bell pepper since its pH was lowered to pH 3.8 within 24 h.

Dielectric properties of salted, acidified vegetables were measured after 4 and 24 h of soaking in a 2% NaCl, 2% citric acid cover solution to monitor the equilibration process. The dielectric constants of each vegetable did not change significantly from their raw values over the 24-h soaking period (Table 1, Fig. 3). In Fig. 3, 0% NaCl refers to ε' measured at time 0 (unsoaked). The datasets labeled as 1% NaCl refer to vegetable pieces soaked in a 2% NaCl solution; i.e., the equilibrated concentration of NaCl in the vegetable pieces was 1%. As expected, dielectric loss factors increased significantly compared to initial values due to ionic losses contributed by NaCl (Fig. 4). This finding was consistent with previous works[10,11] that showed no change in ε' at 915 MHz when sodium chloride was added in the range of 0 to 3% to surimi and cottage cheese, but significant increases in

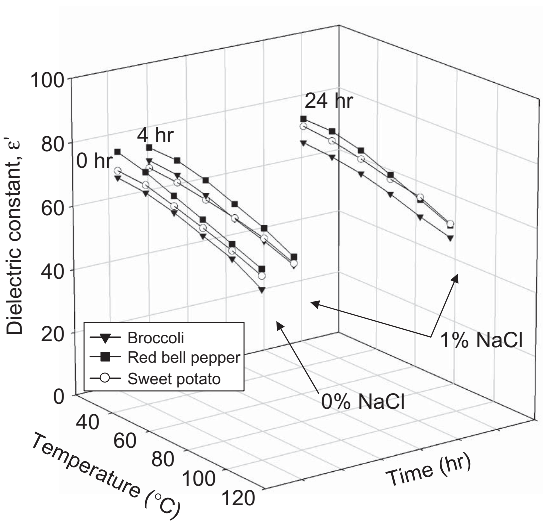

Figure 3 Dielectric constants ε' of broccoli, red bell pepper, and sweet potato measured at 915 MHz as functions of temperature and soaking time (0, 4, and 24 h) in a 2% NaCl (w/v), 2% citric acid (w/v) solution.

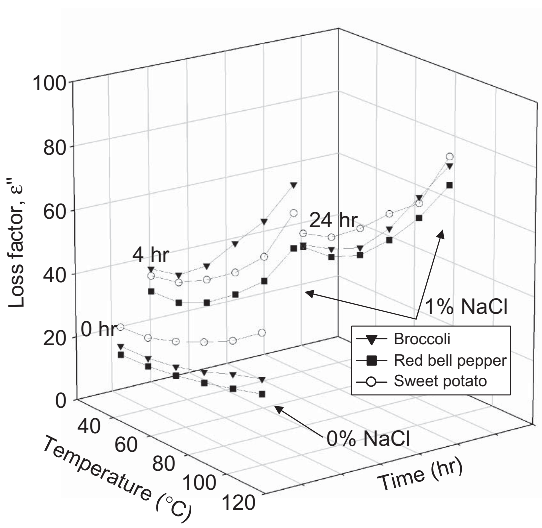

Figure 4 Loss factors ε'' of broccoli, red bell pepper, and sweet potato measured at 915 MHz as functions of temperature and soaking time (0, 4, and 24 h) in a 2% NaCl (w/v), 2% citric acid (w/v) solution.

ε'' were observed and closely related to salt concentration. The current study showed no significant differences in ε'' when the soaking time was increased from 4 to 24 h for each vegetable, and loss factors of different vegetables were not significantly different (Table 1). However, Fig. 4 showed that the difference in loss factors between vegetables was reduced between 4 and 24 h, as shown by the closer grouping of loss factors. Interestingly, Fig. 4 also shows that ε'' of broccoli was greater than sweet potato after 4 h, but not at 0 or 24 h. This observation may indicate that the blanching treatment was more effective in disrupting the cellular structure of broccoli than sweet potato. As a result, the migration of salt into the vegetable tissue occurred more readily in broccoli florets than in sweet potato cubes. This is a logical observation as the florets have more surface area and softer tissue. These results indicated that vegetables should be soaked for about 24 h prior to microwave processing to reduce variability in dielectric properties and subsequent heating by microwaves.

Another useful term used to compare dielectric properties is the loss tangent (tan δ) where tan $\delta = \varepsilon'/\varepsilon''$. The loss tangent provides a better comparison of dielectric properties because it takes into account both ε' and ε''. Comparing tan δ results showed that after soaking, the dielectric properties of the vegetable samples were largely dependent on the salt concentration and similar to a 1% w/v NaCl solution (Fig. 5). This finding suggested that food matrix effects on dielectric properties were minimized when the equilibrated salt content was near 1%. Differences in the loss tangents between vegetables were similar

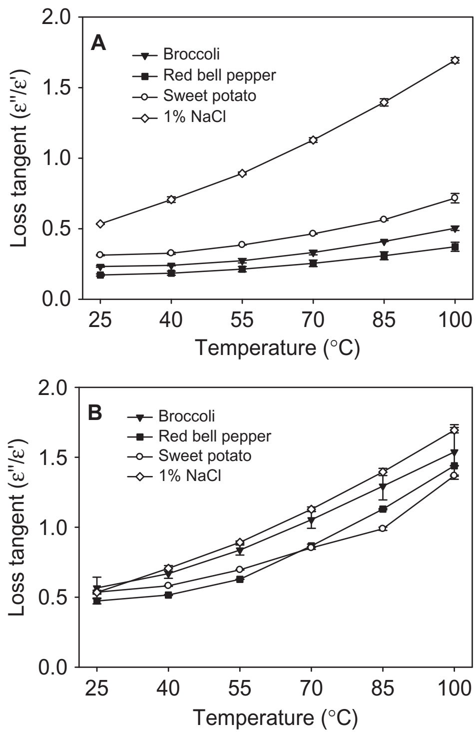

Figure 5 Loss tangents of vegetables before (a) and after (b) soaking in a 2% NaCl, 2% citric acid cover solution for 24 h. Loss tangent of 1% NaCl solution is included as a reference. Error bars represent one standard deviation ($n = 2$).

before and after soaking with salt and acid. However, loss tangent values of all vegetables equilibrated to 1% NaCl were less than a 1% w/v NaCl solution.

While no literature has examined equilibrium phenomena with regards to changes in dielectric properties of salted, acidified vegetables, Sarang and colleagues[4] matched the electrical conductivities of different solid food components by blanching them in a highly conductive sauce prior to ohmic heating, thereby producing uniform heating during ohmic heating. Since electrical conductivity is conferred by the presence of ions, it is closely related to the ionic conductivity term of ε'' as shown by the equation:

$$\varepsilon'' = \varepsilon''_d + \varepsilon''_\sigma = \varepsilon''_d + \frac{\sigma}{\varepsilon_0 \omega},$$

where the subscripts d and σ refer to contributions from dipolar rotation and ionic conductivity, respectively. σ (S m^{-1}) is the ionic conductivity, ε_0 is the permittivity of free space (8.854×10^{-12} F m^{-1}), and ω (rad s^{-1}) is the angular frequency.[12] Guan and others[3] also found salt content, electrical conductivity, and dielectric loss factors to be closely related. It follows that the equilibration of a variety of acidified vegetables to the same salt content would improve heating rates and heating uniformity among vegetable pieces during microwave processing, enabling more efficient heating of a multi-component product.

Effect of Salt Concentration on Dielectric Properties

Vegetables were soaked in cover solutions containing 1 and 2% NaCl to establish the relationship between salt concentration and dielectric properties. The equilibrated salt concentrations in the vegetables were assumed to be 50% of the concentration of salt in the cover solution. After 24 h of soaking, ε' of each vegetable did not change significantly from that of the unsoaked samples ($Pr > F$: 0.9049), regardless of salt concentration (Table 1). However, differences in ε'' with respect to salt concentration were highly significant ($Pr > F$: <0.0001). Figure 6 shows the dielectric loss factors of each vegetable after 24 h soaking periods in 1 and 2% NaCl cover solutions, as well as the unsoaked vegetables. As salt concentration increased, so did ε'' in a linear fashion. Again, this finding was consistent with the work of Herve et al.[10] and Mao et al.,[11] as previously discussed. The addition of more salt increased the ionic conductivity, and produced a positive correlation between salt concentration and dielectric loss.

Effect of Citric Acid Concentration on Dielectric Properties

Different concentrations of citric acid were tested in the presence of NaCl to determine the importance of acidulant levels on the dielectric properties of acidified vegetables. For this experiment, the salt concentration was held constant, and the citric acid concentrations varied according to each vegetable. Treatment 1 involved soaking the vegetables in cover solutions containing 2% NaCl and 2% citric acid. For comparison, treatment 2 involved soaking each vegetable in a cover solution containing 2% NaCl and their titration-determined amount of citric acid required to lower the tissue pH to 3.8 (Fig. 1). For red bell pepper, sweet potato, and broccoli these amounts were 0.5, 1.0, and 1.5% w/v citric acid, respectively.

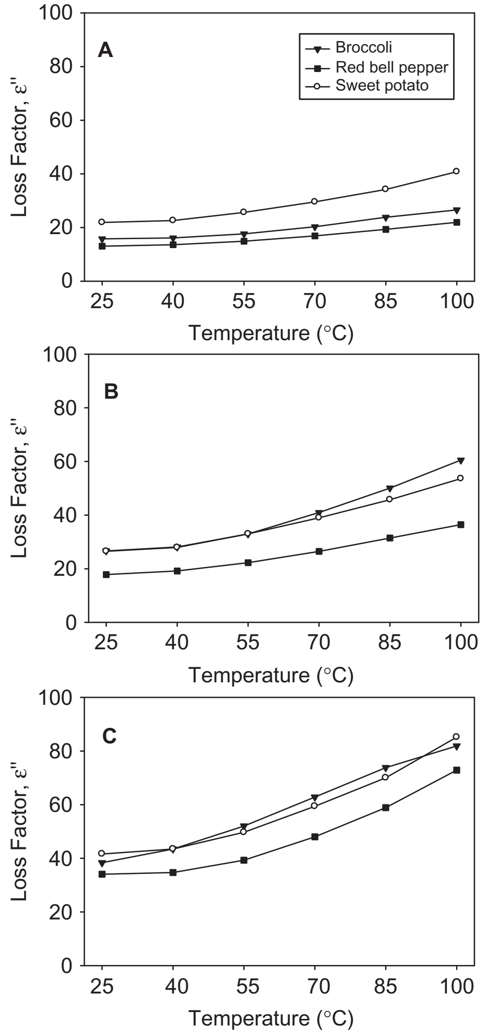

Figure 6 Effect of salting on dielectric loss factor ε'' of broccoli, red bell pepper, and sweet potato acidified with its titration-determined level of citric acid: (a) no salt, 0 h, and (b) 24 h soaking period in 1% NaCl and (c) 2% NaCl cover solutions.

Citric acid concentrations ranging from 0.5–2.0% w/v in the cover solution had no significant effect on ε', but had a slight effect on ε'' (Pr > F: 0.0615) when 2% NaCl was present in the cover solution (Table 1). Figure 7 shows this effect graphically by examining the loss factors of each vegetable at their respective citric acid concentrations compared to all vegetables at the same citric acid concentration of 2%. The minimal contribution of citric acid to the dielectric properties of the vegetables was likely due to its molecular structure and the narrow range of concentrations tested. The degree of interaction of a molecule to a microwave field depends on its size, polarity, and conductivity.[13,14] Small, highly conductive ionic compounds, such as NaCl, readily dissociate in solution and are

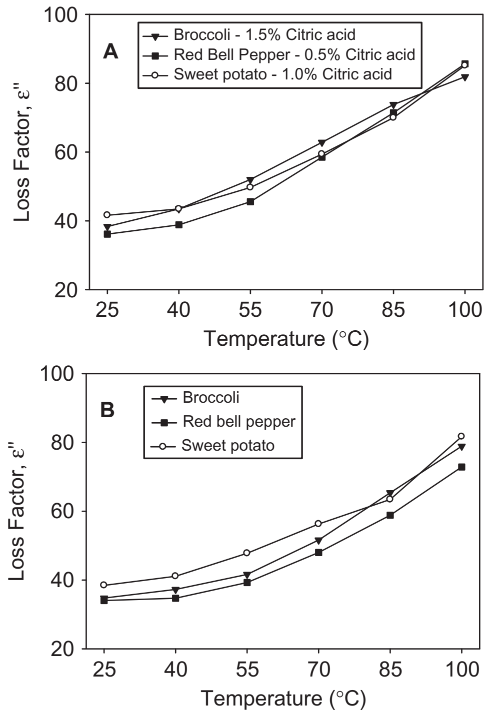

Figure 7 Dielectric loss factors ε'' of vegetables after 24 h soaking period in a 2% w/v NaCl solution with titration-determined citric acid concentrations (a), and constant citric acid concentration of 2% w/v (b).

very mobile when exposed to 915 MHz microwaves. Citric acid is a larger molecule and has only one charged carboxyl group at pH 3.8. Therefore, due to its lower charge and larger size, citric acid cannot respond as readily to the oscillating electric field, so its contribution to dielectric loss is less. Therefore, when both NaCl and citric acid were present in the system, the contribution of NaCl overshadowed any measureable effect of citric acid. These results showed that concentrations of citric acid typically used in acidified vegetables had little effect on the dielectric properties when vegetables were soaked in a cover solution containing 2% NaCl.

CONCLUSION

This research was consistent with previous studies and showed that salt was the major contributor to changes in dielectric properties of acidified vegetable products. Salt concentration had no significant effect on the dielectric constant of acidified vegetables, but was significantly associated with the dielectric loss factor. Variability in dielectric properties decreased as soaking time increased. Citric acid was found to have minimal effects on the dielectric properties of acidified vegetables when used in conjunction with sodium chloride at levels of 0.5–2.0%. Furthermore, these findings provide food formulators flexibility in acidulant levels when designing foods for microwave processing.

REFERENCES

1. Thostenson, E.T.; Chou, T-W. Microwave processing: Fundamentals and applications. Composites Part A **1999**, *30*, 1055–1071.
2. Nelson, S.O.; Datta, A.K. Dielectric properties of food materials and electric field interactions. In: *Handbook of Microwave Technology for Food Application*; Datta, A.K.; Anantheswaran, R.C.; Eds.; Marcel Dekker: New York, 2001; 61–114.
3. Guan, D.; Cheng, M.; Wang, Y.; Tang, J. Dielectric properties of mashed potatoes relevant to microwave and radio-frequency pasteurization and sterilization processes. Journal of Food Science **2004**, *69* (1), E30–E37.
4. Sarang, S.; Sastry, S.K.; Gaines, J.; Yang, T.C.S.; Dunne, P. Product formulation for ohmic heating: Blanching as a pretreatment method to improve uniformity in heating of solid-liquid food mixtures. Journal of Food Science **2007**, *72* (5), 227–234.
5. Tanaka, F.; Morita, K.; Mallikarjunan, P.; Hung, Y.-C.; Ezeike, G.O.I. Analysis of dielectric properties of rice vinegar and sake. Transactions of the American Society of Agricultural Engineers **2002**, *45* (3), 733–740.
6. Bohigas, X.; Tejada, J. Dielectric properties of acetic acid and vinegar in the microwave frequencies range 1–20 GHz. Journal of Food Engineering **2009**, *94*, 46–51.
7. Lau, M.H.; Tang, J. Pasteurization of pickled asparagus using 915 MHz microwaves. Journal of Food Engineering **2002**, *51*, 283–290.
8. Kumar, P.; Coronel, P.; Simunovic, J.; Truong, V.D.; Sandeep, K.P. Measurement of dielectric properties of pumpable food materials under static and continuous flow conditions. Journal of Food Science **2007**, *72* (4), E177–E183.
9. Brinley, T.A.; Truong, V.D.; Coronel, P.; Simunovic, J.; Sandeep, K.P. Dielectric properties of sweet potato purees at 915 MHz as affected by temperature and composition. International Journal of Food Properties **2008**, *11*, 158–172.
10. Herve, A.-G.; Tang, J.; Luedecke, L.; Feng, H. Dielectric properties of cottage cheese and surface treatment using microwaves. Journal of Food Engineering **1998**, *37*, 389–410.
11. Mao, W.; Watanabe, M.; Sakai, N. Dielectric properties of Surimi at 915 MHz and 2450 MHz as affected by temperature, salt and starch. Fisheries Science **2003**, *69*, 1042–1047.
12. Tang, J. Dielectric properties of foods. In: *The Microwave Processing of Foods*; Schubert, H.; Regier, M.; Eds.; CRC Press: Boca Raton, 2005; 22–40.
13. Gabriel, C.; Gabriel, S.; Grant, E.H.; Halsted, B.S.J.; Mingos, D.M.P. Dielectric parameters relevant to microwave dielectric heating. Chemical Society Review **1998**, *27*, 213–223.
14. De Los Reyes, R.; Heredia, A.; Fito, P.; De Los Reyes, E.; Andres, A. Dielectric spectroscopy of osmotic solutions and osmotically dehydrated tomato products. Journal of Food Engineering **2007**, *80*, 1218–1225.