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ASCORBIC ACID DEGRADATION KINETICS AND MASS TRANSFER EFFECTS IN BIOLOGICAL TISSUE DURING OHMIC HEATING

DISSERTATION

Presented in Partial Fulfillment of the Requirements for the Degree Doctor of Philosophy in the Graduate School of The Ohio State University

By

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The Ohio State University
1996

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ABSTRACT

Ohmic or electrical heating is of interest in food processing, and exhibits numerous novel phenomena not seen with conventional heating. To more fully characterize features of ohmic heating, degradation kinetics of ascorbic acid and mass transfer of components were studied. Ascorbic acid degradation kinetics in orange juice in the temperature range of 65°-90°C was unaffected by an electric field strength of 18.2 V/cm. Ohmic heating resulted in significantly greater diffusion of dye from beetroot at 42° and 58°C, but not at 72°C. The electrical conductivity of beetroot was higher during ohmic heating than conventional heating when diffusion enhancement was observed. The influence of frequency and wave form of alternating current on the electrical conductivity and heating rate of turnip tissue was investigated. The electrical conductivity was significantly higher for sine and triangular waves at 4 Hz., but not at 10, 25 or 60 Hz. No significant variation in electrical conductivity was observed from 4-60 Hz. using the square wave. The heating rate increased as frequency decreased. The effects of ohmic heating frequency on hot-air drying rate, desorption isotherms, rehydration of dried samples, and juice yield was studied. The highest drying rates and juice yields were obtained using a 4 Hz. frequency of alternating current. Ohmic heating exhibited several novel effects, including tissue softening and an increase in sample surface moisture at relatively low temperatures, and
an increased integrity of color when held at low relative humidities over time. Using ohmic heating at low frequencies to enhance the diffusion of specific components appears to be a useful method for processes involving mass transfer.
This work is dedicated to Lynn E. Hathaway

Every woman I have ever loved has left her print upon me, where I loved some invaluable piece of myself apart from me—so different that I had to stretch and grow in order to recognize her. And in that growing, we came to separation, that place where work begins. Another meeting.

Audre Lorde
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and told me I was going to be okay. I had the good sense to believe her; it was easy to do with all her nurturing support. Sue’s zest for life is infectious, her capacity for caring tremendous.

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Agricultural Engineering
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CHAPTER 1

INTRODUCTION

Ohmic or electrical heating involves the passage of an alternating current through a sample. The sample responds by generating heat internally due to its inherent resistance. This is of interest in food processing because of the potential to uniformly heat large particulates at comparable rates with liquid carrier fluids, a scenario that results in superior food quality. Several other novel effects that occur during ohmic heating are receiving increased attention. In this work, several foods are ohmically heated and corresponding parameters of interest examined.

Much research concerning ohmic heating has centered on heat transfer issues. Models have been developed to predict the temperature distribution of single particles, and liquid-particle mixtures during ohmic heating in static and continuous situations (de Alwis and Fryer, 1990; Sastry and Palaniappan, 1992; Sastry, 1992). One issue worthy of study is degradation kinetics of vitamins during ohmic heating. It is well established that the high temperatures used in food processing are conducive to the breakdown of vitamins. By comparing degradation kinetics of vitamins during ohmic heating with those occurring in
an established conventional process, it is possible to determine what influence an electric field has on nutrient degradation.

Ohmic heating is critically dependent on the electrical conductivity (σ), which is influenced by food chemistry and the type of heating. The electrical conductivity increases with increasing temperature, applied voltage, concentration of electrolytes (Palaniappan and Sastry, 1991a,b), particle size and pretreatments such as soaking. Halden et al. (1990) reported that starch transition, phase change and cell wall break down affect σ during ohmic heating. All studies characterizing σ during ohmic heating conditions were conducted using alternating current at 50 or 60 Hz with a sinusoidal wave form, the standard output for the United Kingdom and the United States respectively. Recent studies (Imai et al., 1995, Kim and Pyun, 1995) have suggested that changing the frequency and wave form of alternating current applied to foods influences the heating rate. The influence of frequency and wave form on electrical conductivity has not been investigated.

While heat transfer properties have been well studied, additional effects involving mass transfer occur during ohmic heating that are not seen with conventional processes. Wigerstrom (1976) showed that electric fields enhanced moisture loss during the blanching of potato slices. Katrokha et al. (1984) used an electric field to intensify the extraction of sucrose from sugar beets. Halden et al. (1990) observed an increase in the diffusion of beet dye from beetroot into a carrier fluid during ohmic heating and suggested that electro-osmosis was responsible. This work was expanded by Schreier et al. (1993), who
estimated the extent of diffusion enhancement for beetroot during ohmic heating from 20-80°C. Most recently, ohmic heating has been used to aid the extraction of soy milk from soybeans (Kim and Pyun, 1995).

These mass transfer effects seen during ohmic heating could have widespread utility. Wang (1995) determined that ohmic heating was an effective pretreatment for improving drying, and apple juice extraction. Additional improvements in mass transfer operations may be possible by adjusting the frequency and wave form of alternating current applied to the food sample.

The objectives of this work were:

1. To determine if an electric field altered the rate of vitamin degradation kinetics during ohmic heating.

2. To examine differences in diffusion between conventional and ohmic heating at steady-state temperature.

3. To investigate the effect of frequency and wave form of alternating current on electrical conductivity.

4. To determine how the frequency and wave form of alternating current affects mass transfer properties for food processing unit operations.
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CHAPTER 2

ASCORBIC ACID DEGRADATION KINETICS DURING CONVENTIONAL AND OHMIC HEATING

ABSTRACT

Ascorbic acid (vitamin C) degradation during electrical and conventional heating was studied to determine if the presence of an electric field altered the rate of degradation. Experiments were performed using a static ohmic heating apparatus, and heating histories were matched using applied voltage in the electrical case and hot oil in the conventional case. A statistical analysis showed that electric field has no significant effect on ascorbic acid degradation. Pseudo first order degradation kinetics were observed for both conventional and ohmic cases. The effect of electrolysis on ascorbic acid degradation was also examined. Gas production and dissolution appeared to occur with stainless steel electrodes, and was not evident with specially coated titanium electrodes. These phenomena did not affect ascorbic acid activity.
INTRODUCTION

Ohmic or electrical heating shows considerable promise as a viable sterilization option (Stirling, 1987). Parrot and Palaniappan and Sastry (1992) found that under certain circumstances, large food particulates and carrier fluids can heat at comparable rates during ohmic heating. This very desirable scenario is impossible to achieve using conventional heating. However, critical safety issues must be addressed before this technology meets regulatory approval.

It is well established that high temperatures are conducive to the breakdown of vitamins. Numerous studies have been conducted to quantify the breakdown of ascorbic acid at elevated temperatures. The degradation kinetics of ascorbic acid in fruit juices using a 20° to 92°C range was studied by dio Alavarado and Viteri (1991). They used two types of orange juice and found both to follow first order kinetics, with activation energies of 9.26 kcal/mol and 11.1 kcal/mol respectively. Corresponding frequency factors were $1.3 \times 10^4$ min$^{-1}$ and $3.7 \times 10^5$ min$^{-1}$. Nagy and Smoot (1977) and Nagy (1980) investigated temperature and storage effects on percent retention of ascorbic acid in orange juice. They found that first order degradation kinetics were followed at 29°C but not at 38° or 46°C. The same investigators also determined that the Arrhenius relationship was observed in a 4 to 50°C temperature range, but had two distinct profiles at 4 to 28°C and 28 to 50°C. The activation energy for the former range was reported as 12.8 kcal/mol, and 24.5 kcal/mol for the latter. Frequency factors were not reported.
While kinetic data are available for ascorbic acid degradation in orange juice using conventional heating, kinetic data have not yet been determined for ascorbic acid degradation under ohmic conditions.

Another question that merits attention is the potential for electrolysis. Electrochemical phenomena can result in gas liberation at the electrodes and the release of metal ions into solution (Castellan, 1983), both of which could affect vitamin activity. Of particular interest in this study were uncoated stainless steel and specially coated titanium electrodes (coating provided by APV Company, Devon, England).

Accordingly, the objectives of this study were (1) to determine the effect of the presence of an electric field on the rate of ascorbic acid degradation in orange juice, (2) to determine the kinetic parameters under conventional and ohmic conditions and (3) to test for evidence of electrolysis using stainless steel and specially coated titanium electrodes during ohmic heating.

MATERIALS AND METHODS

Samples of strained orange juice (preparation described below) were subjected to conventional and ohmic heating using identical time-temperature histories in each case. Four time-temperature combinations were used: (1) holding at 65°C for one hour, with 0.5 mL samples taken after 0, 20, 40 and 60 minutes; (2) holding at 75°C for forty-five minutes with 0.5 mL samples taken after 0, 15, 30 and 45 minutes; (3) holding at 80°C
with 0.5 mL samples taken after 0, 10, 20 and 30 minutes and (4) holding at 90°C with 0.5 mL samples taken after 0, 10, 20 and 30 minutes.

Ascorbic acid concentrations were determined using the method outlined by Howard et al. (1987), and analysis of variance was performed on percent degradation of ascorbic acid activity.

Evidence of electrolysis was studied using the following methods: (1) the ohmic heater was observed to determine if gas liberation at the electrodes was visible during ohmic heating. This observation was conducted both with orange juice as well as 0.22% salt solution to facilitate better observation. Careful observations were made for the presence of rust on the electrodes. (2) The orange juice was tested to determine if iron was present in solution, with the presence of a greater amount of iron in solution after ohmic heating being considered an indicator of electrolysis. (3) Ascorbic acid concentration was determined by sampling from the center of the ohmic heater at all times. After final sampling, the heater was shaken to thoroughly mix the contents, then another sample was taken immediately. The hypothesis was that if electrolysis resulted in a decreased concentration of ascorbic acid in the region immediately adjacent to the electrodes, one might expect to find a lower concentration of ascorbic acid in the overall sample after shaking. This might be too small for the instrument to detect, especially if the chamber volume to electrode area ratio is large.
Apparatus

The apparatus shown in Figure 2.1 was used in both conventional and ohmic cases, and consisted of a Pyrex glass tee (inside diameter 2.54 cm.), the ends of which were equipped with electrodes. The electrodes were held in place by a metal flange with a rubber gasket to prevent leakage of constituents. When properly tightened, a 6.6 centimeter gap existed between electrodes.

A rubber stopper at the top of the tee was equipped with sampling and thermocouple ports, both of which were sealed to prevent evaporation and exposure of the sample to air. Samples were withdrawn using B-D 3 cc Luer-lock syringes (Fisher, Pittsburgh, PA) equipped with 18 gauge needles.
Sample preparation

The sample used for this study was pasteurized orange juice (Tropicana, pure premium). The expiration date on the container was at least four weeks after the last day experiments were conducted, and batches of orange juice used were not kept longer than five days in order to ensure relative uniformity of the sample. The juice was stored at 4°C in a light tight plastic container. The container was gently shaken in the same manner whenever a sample was withdrawn to ensure as homogenous a sample as possible. Subsequently, the orange juice was strained through cheese cloth to remove pulp and other solids.

A 45 mL aliquot of strained orange juice was placed in the heater apparatus, which was then immersed in a 25°C water bath and allowed to equilibrate (approximately 15 minutes).

Matching of time-temperature histories

Precise matching of time-temperature histories in conventional and ohmic cases was considered necessary to eliminate temperature as a variable. Thus, it became necessary to ensure that the measured temperature was representative of the average temperature throughout the heater.

Studies were undertaken to determine the extent of thermal gradients existing within the heater in each case. The liquid crystal method used by Balasubramaniam and Sastry (1994) was employed to determine the temperature distribution in the heater at any
time. Since visual observation was necessary, we used a 0.22% salt solution of electrical conductivity which matched that of the orange juice over the temperature range.

During ohmic heating, the average heater temperature lagged the center temperature approximately 2°-3°C. In the conventional case, the average heater temperature led the center temperature 3°-5°C. These phenomena can be viewed in Figure 2.2, and are due to the inside-to-outside heating representative of ohmic heating (internally generated), and the outside-to-inside heating characteristic of conventional heating. These discrepancies were corrected by matching the average heater temperature for both conventional and ohmic cases. During conventional heating, the lag between the center and average temperatures closed when approaching the holding temperature due to agitation created by the heater being removed from the oil bath and held in air for several seconds before being placed in the holding bath. Typical temperature distributions in the heater for conventional and ohmic heating are shown in Figure 2.3.

Exact matches were employed for the 65°, 75° and 80°C temperatures. The 75°C match is shown in Figure 2.4. The 90°C point could not be matched using the liquid crystal method, as this was out of the measurement range of the liquid crystal. However, similar trends present in the 65°, 75° and 80°C data were extended to the 90°C case.
Figure 2.2 Center vs. average temperature profiles for ohmic and conventional heating
Figure 2.3. Temperature distributions (°C) in heater

(a) ohmic

T_{\text{average}} = 42.9 °C
T_{\text{center}} = 44.4 °C

(b) conventional

T_{\text{average}} = 47.5 °C
T_{\text{center}} = 44.0 °C
Figure 2.4. Comparison of conventional and ohmic time-temperature histories at 75 °C
Experimental setup

For ohmic heating conditions, the heater was immersed in a 25°C water bath. The power was turned on, and the sample subjected to an 18.2 V/cm electric field. Current reached a maximum of 1.06 amperes during the heating portion of the experiments. Upon approaching the holding temperature, the voltage was ramped down to approximately 80% of the initial value (120 V). This was sufficient to hold the sample at the desired temperature.

For conventional conditions, the heater was immersed in a 120°C oil bath. Upon approaching the holding temperature, the heater was transferred to a water bath at the holding temperature.

Sampling

Samples of 0.5 mL were withdrawn in the manner described above. A protective plastic sheath was placed over the needle as soon as the sample was removed from the heater, and the syringe and protected needle were wrapped in aluminum foil and placed on ice immediately. Samples were kept on ice and in the absence of light until assayed for ascorbic acid content.

Interference between the metal needle and the electric field was tested by checking for fluctuations in temperature, voltage and current measurements when the needle was in direct contact with the electric field. No fluctuations were observed during the sampling time (approximately three seconds).
Assays

1. Ascorbic acid. Ascorbic acid concentration was determined using High-Performance Liquid Chromatography (HPLC), following the method of Howard et al. (1987) after modifications. A liquid chromatograph equipped with an injector valve and a spectrophotometric detector set at 244 nm was used. An integrator was used to calculate peak areas. Separation was achieved with a C_{18} column. The mobile phase was methanol-HPLC grade water (10:90) with 0.1% phosphoric acid; this solution was filtered and de-gassed via vacuum before being used on the column. A flow rate of 1.2 mL/min was employed, and retention time for ascorbic acid was 2.75 minutes.

A standard calibration curve was made using L-ascorbic acid (Sigma, St. Louis, MO) and mobile phase. Curves were generated each time the HPLC was used. Samples were taken from ice and diluted 1:1 in mobile phase. This mixture was taken up in a one mL TB syringe (Fisher, Pittsburgh, PA) and filtered through a 0.45 μm Acrodisc filter (Fisher, Pittsburgh, PA). Samples were measured in triplicate.


3. pH. The pH was measured using a Beckman pH meter, and was calibrated using pH 4 and pH 7 standards.

Statistical analysis

All statistical analyses were performed by the SAS statistical analysis system, and data were analyzed using the general linear model. Percent ascorbic acid degradation at
the end of the heating time was the dependent variable, while treatment (type of heating) and temperature were the class variables. For the two treatments (conventional and ohmic), four time-temperature schemes were used, and five to six replications for each of the eight combinations were taken. The study was completely randomized with respect to order of experimentation. Kinetic constants were determined using a linear regression model. Three repetitions were used to generate kinetic constants, because during the first half of the experiment described above, only percent degradation data were taken.

RESULTS AND DISCUSSION

The mean percent ascorbic acid degradation for each case can be seen in Table 2.1.

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<th>Temperature, °C</th>
<th>Type of heating</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ohmic</td>
</tr>
<tr>
<td>65</td>
<td>13.1a</td>
</tr>
<tr>
<td>75</td>
<td>11.3a</td>
</tr>
<tr>
<td>80</td>
<td>12.9a</td>
</tr>
<tr>
<td>90</td>
<td>21.2b</td>
</tr>
</tbody>
</table>

Table 2.1. Percent degradation of ascorbic acid activity in orange juice (mean values)

a, b values followed by the same letter are not significantly different (P ≤ 0.05)
Mean comparisons for percent degradation of ascorbic acid in orange juice under conventional and ohmic conditions showed that the type of heating has no statistically significant effect. Vitamin C degradation was a function of temperature and the linear model procedure indicated that the 90°C treatment was different from the 65°, 75° and 80°C treatments. The degradations were comparable at 65°, 75° and 80°C due to the differences in the amount of time the sample was exposed to the set point temperature. The interaction terms were not significant, indicating that the temperature effect does not influence the treatment effect. Sample pH was measured and found to remain constant at 3.8 before and after both conventional and ohmic heating, thus it does not appear to change the reaction mechanism in either mode of heating.

Ascorbic acid degradation followed pseudo first order kinetics for both conventional and ohmic heating. Rate constants \( (k) \) for the conventional and ohmic cases were plotted vs. the reciprocal of absolute temperature to determine frequency factor \( (A) \) and activation energy \( (E_a) \). These Arrhenius plots are shown in Figure 2.5, and corresponding kinetic parameters are listed in Table 2.2.
Figure 2.5. Arrhenius plots for conventional and ohmic heating

Table 2.2. Kinetic constants for ascorbic acid degradation in orange juice

<table>
<thead>
<tr>
<th>Type of heating</th>
<th>$A$, min$^{-1}$</th>
<th>$E_a$, kcal/mol</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>$19.95 \times 10^3$</td>
<td>12.6</td>
<td>0.98</td>
</tr>
<tr>
<td>Ohmic</td>
<td>$19.59 \times 10^3$</td>
<td>12.5</td>
<td>0.91</td>
</tr>
</tbody>
</table>
The measured kinetic constants are comparable to published literature values, although activation energy is more widely reported than frequency factor. Values from various studies are shown in Table 2.3.

<table>
<thead>
<tr>
<th>Investigators</th>
<th>Order of reaction</th>
<th>$E_a$, kcal/mol</th>
<th>$A$, min$^{-1}$</th>
<th>Temperature range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nagy and Smoot</td>
<td>1</td>
<td>12.8</td>
<td>NR</td>
<td>4 - 28° C</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>24.5</td>
<td>NR</td>
<td>28 - 50° C</td>
</tr>
<tr>
<td>Alvarado and Viteri</td>
<td>1</td>
<td>11.1</td>
<td>$3.7 \times 10^5$</td>
<td>20 - 92° C</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>9.3</td>
<td>$1.3 \times 10^4$</td>
<td>20 - 92° C</td>
</tr>
<tr>
<td>Laing et al.</td>
<td>0</td>
<td>14 - 17</td>
<td>NR</td>
<td>61 - 105° C</td>
</tr>
<tr>
<td>Kaanane et al.</td>
<td>0</td>
<td>13.3</td>
<td>NR</td>
<td>4 - 45° C</td>
</tr>
</tbody>
</table>

Table 2.3. Selected literature values of kinetic constants obtained for ascorbic acid degradation in orange juice (NR = not reported)

The activation energies obtained in this study are in the range of reported literature values for conventional heating. The values reported for frequency factor are higher than those reported by Alvarado and Viteri (1991), but are reasonably close to these data.

Tests for electrolysis

There was noticeable gas liberation (cavitation) at the stainless steel electrodes, and no visible liberation with the specially coated titanium electrodes. The effect was especially pronounced with orange juice; the 0.22% salt solution with stainless steel...
electrodes did not exhibit cavitation, but bubbles covered the entire electrode surface during ohmic heating. No bubbles were observed on the titanium electrodes using 0.22% salt solution.

The salt solution was subjected to identical ohmic treatments using the stainless steel and specially coated titanium electrodes. After heating, there was a noticeable rust tinge in the solution with stainless steel electrodes, and no tinge with the titanium electrodes. When disassembled, the stainless steel electrode surfaces were covered with rust. No residue was present on the titanium electrode surfaces.

The AOAC iron method was used to test for the presence of iron in orange juice before and after ohmic heating using the stainless steel electrodes and the 90°C heat treatment. Iron, a constituent of stainless steel, could be present in solution if metal dissolution occurs. A trace of iron (<4 ppm) was found in the orange juice before ohmic heating, and an iron concentration of 40 ppm was found after heating. This suggests that metal corrosion occurs with stainless steel electrodes. No comparable test was performed on the coated titanium electrodes, since the target material (from the coating) was unknown.

The final test for electrolysis yielded no difference in ascorbic acid concentration before and after mixing for both sets of electrodes. This indicates that any phenomena near the electrodes were not sufficiently pronounced to provide a bulk change in ascorbic acid concentration. Because the volume to electrode surface area ratio was relatively large (6.94 mL/cm²), this test may not be very sensitive. From these tests, it appears that visible
gas production does not occur using the coated electrodes and does occur with the stainless steel electrodes. We did not attempt to detect any increase in soluble gas concentrations. Metal dissolution does not appear to be visible with coated titanium electrodes, but is evident with stainless steel electrodes.

The 80°C treatment was performed in duplicate using both sets of electrodes to determine if electrolysis affected the rate of ascorbic acid degradation, and no difference was observed. This indicates that although both gas liberation and corrosion appear to be occurring with stainless steel electrodes, they do not affect the rate of ascorbic acid degradation. The presence of iron can adversely affect ascorbic acid activity (Tannenbaum et al., 1985) by catalyzing degradative pathways in the presence of oxygen. However, ascorbic acid degradation in foods is a complex function of many variables, including temperature, pH, oxygen concentration, salt and sugar concentration, and the presence of enzymes and metal catalysts. Oxygen concentration in orange juice may not be a major factor due to the presence of solutes, which have a salting-out effect on dissolved oxygen. Thus, while iron may be present in solution, high solute concentrations in orange juice may preclude any adverse effects caused by iron.

Ascorbic acid degradation during ohmic heating as a function of temperature, pH level, NaCl concentration, electrode type and applied power was studied by Assiry (1996). This investigator found that ascorbic acid degradation kinetics were pseudo first order for both conventional and ohmic heating conditions. Activation energies were lower than those found here, probably due to the difference in the system studied (he used buffer
solutions with NaCl and ascorbic acid added). In Assiry's study (1996), the more corrosion that was observed, the less the ascorbic acid degradation. Assiry (1996) hypothesized that the corroded metal material competes with ascorbic acid for dissolved oxygen. A higher degradation rate was obtained during ohmic heating with specially coated titanium electrodes as compared to conventional heating under similar environmental conditions. The difference between Assiry's result and the one obtained here is probably due to the difference in the system studied. Dissolved oxygen has a less pronounced effect in orange juice because of the salting-out effect discussed above. Thus, oxygen produced during ohmic heating with titanium coated electrodes will have a more exaggerated effect on the buffer system which contains only NaCl.
CONCLUSIONS

Ascorbic acid degradation kinetics in orange juice in the temperature range of 65°-90°C was unaffected by an electric field of strength 18.2 V/cm. Ascorbic acid degradation followed first order kinetics for both conventional and ohmic treatments, and kinetic constants were in the range of reported literature values. Electrolytic reactions (both gas production and dissolution) appear to occur when using stainless steel electrodes, but are not evident with specially coated titanium electrodes. No difference in percent ascorbic acid degradation was observed using either set of electrodes.
NOTATION

A               frequency factor, min\(^{-1}\)
ANOVA           analysis of variance
AOAC           Association of Official Analytical Chemists
E\(_a\)         activation energy, kcal/mol
HPLC           high-performance liquid chromatograph
k               rate constant, min\(^{-1}\)
T               Temperature, K
ln              natural log
R\(^2\)         coefficient of variation
V               volts
REFERENCES


CHAPTER 3

DIFFUSION OF BEET DYE DURING ELECTRICAL AND CONVENTIONAL HEATING AT STEADY-STATE TEMPERATURE

ABSTRACT

Diffusion of dye from beetroot tissue into a fluid was studied during conventional and ohmic heating as a function of steady-state temperature. The volume of beet dye diffusing into solution during ohmic heating was enhanced with respect to conventional heating at 42°C and 58°C, but not at 72°C. This can be explained by examining the differences in electrical conductivity of beet tissue at these temperatures during conventional and ohmic heating. At 42°C and 58°C, the electrical conductivity of beet tissue heated ohmically is higher than the electrical conductivity of beet tissue heated conventionally. At 72°C, the electrical conductivities of beet tissue during conventional and ohmic heating are equal. The extent of diffusion in the ohmic case is also positively correlated with applied voltage.
INTRODUCTION

In ohmic heating, alternating current is passed through food, which is heated due to its inherent electrical resistance. The primary property affecting ohmic heating is the electrical conductivity of the food (Sastry and Palaniappan, 1992). Electrical conductivity (σ) is a function mainly of food chemistry and structure, and temperature (T). The presence of ionic substances such as acids and salts enhances σ, while the presence of non-polar constituents like fats and lipids decreases it (Wang and Sastry, 1993, Sastry and Palaniappan, 1992). Because the heat generation rate obtained during ohmic heating is proportional to the electrical conductivity at a constant voltage gradient, this property has direct implications on heat transfer.

Relatively few studies exist concerning mass transfer during ohmic heating. Wigerstrom (1976) found that electric fields enhanced moisture loss during the blanching of potato slices, and Katrokha (1984) used electric heating to aid the extraction of sucrose from sugar beets. Kim and Pyun (1995) examined the extraction of soy milk from soybeans using ohmic heating. Schreier et al. (1993) investigated the diffusion of beetroot dye from beetroot, and Rhodamine B dye from semipermeable tubing. The sample was heated from 20° to 80°C and the amount of beet dye diffusing into solution was measured. However, because identical time-temperature histories for conventional and ohmic treatments were used, continuous ohmic heating was impossible. Schreier et al. (1993) showed a 40% enhanced diffusion effect for ohmic heating, and that the concentration of
dye was directly proportional to beetroot particle surface area, and a linear function of electric field strength. This study was motivated by our interest in understanding electrical diffusion enhancement under a continuous electric field, and under constant temperature.

Thus, the objective of this study was to compare the extent of diffusion at constant temperature between continuous ohmic heating and conventional heating.

MATERIALS AND METHODS

The amount of beet dye liberated from a 1.15 cm. cube of beet tissue was determined for three temperatures, (42°C, 58°C and 72°C), two carrier fluids (0.1% and 0.2% NaCl, with electrical conductivities 0.17 S/m and 0.30 S/m respectively) and two heating treatments, conventional and ohmic. For all trials, 50 mL of the carrier fluid was poured into the heater (details below), resulting in a 2 cm. depth of solution in the heater. The entire apparatus was placed into a shaker bath and the sample was introduced through the sampling port. The heater was constantly agitated at 137 rpm; after three minutes, the sample was removed from the carrier fluid, and the optical density of this solution measured at 527 nm with a spectrophotometer (Beckman DU-50, Fullerton, CA). The volume of beet dye liberated from the sample was determined using a standard curve (see details below).
**Apparatus.** The ohmic heater consisted of a 250 mL beaker with titanium electrodes connected to a 120 V, 60 Hz. AC power supply as shown in Figure 3.1. A plastic cap held the electrodes in place, and was equipped with a sampling port and a thermocouple port. A datalogger (Campbell Scientific, Logan, UT) was used to monitor voltage, current and temperature of the fluid on a continuous basis. Temperature was measured using a Teflon coated type T thermocouple.

![Figure 3.1. Experimental apparatus](image-url)
Temperature control. An isothermal condition was important in these studies. Initial ohmic trials with no agitation showed a temperature gradient of 4.3°C per centimeter in the vertical direction (0°C in the axial direction). Using a shaker speed of 137 rpm, the solution had a 0.75°C per centimeter temperature gradient in the vertical direction. The mean temperature was used as the isothermal temperature. To maintain an isothermal environment during the three minute test period during ohmic trials, it was necessary to slightly ramp down the applied voltage. Table 3.1 shows voltage-temperature combinations.

<table>
<thead>
<tr>
<th>Carrier Fluid σ, S/m</th>
<th>Heater Temperature, °C</th>
<th>Water Bath Temperature, °C</th>
<th>Applied Voltage, V</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.17</td>
<td>42</td>
<td>25</td>
<td>107±2</td>
</tr>
<tr>
<td></td>
<td>58</td>
<td>35</td>
<td>113±2.5</td>
</tr>
<tr>
<td></td>
<td>72</td>
<td>48</td>
<td>115±5</td>
</tr>
<tr>
<td>0.3</td>
<td>42</td>
<td>25</td>
<td>75±1.5</td>
</tr>
<tr>
<td></td>
<td>58</td>
<td>35</td>
<td>96±3</td>
</tr>
<tr>
<td></td>
<td>72</td>
<td>48</td>
<td>102±6</td>
</tr>
</tbody>
</table>

Table 3.1. Experimental parameters for ohmic heating trials

For conventional heating trials, the shaker bath was heated to the appropriate temperature and the ohmic heater with carrier fluid was loaded into the bath. Experiments commenced when the temperature of the carrier fluid was at thermal equilibrium with the temperature of the shaker bath.
Standard curve. Standard curves were used to determine the volume of beet dye diffusing from the sample. Preparation consisted of dissolving a known volume of pure beet juice (extracted mechanically from beets) into a known volume of carrier fluid and measuring the optical density of the solution at 527 nm. Separate curves were made for the two carrier solutions, see Figure 3.2.

![Figure 3.2. Standard curve](image-url)
Sample preparation and statistics. Beets obtained from the same vendor were cut into cubes with razor blades immediately prior to immersion in the heater. A minimum of six repetitions were performed for each combination of type of heating (conventional or ohmic), temperature, and carrier fluid electrical conductivity. Two to three beets were used to obtain this many repetitions for a comparison between treatments (for example, one beet usually yielded three repetitions of conventional and ohmic treatments for one temperature and electrical conductivity). Because the amount of dye liberated varies from beet to beet, the standard deviations for this study may be higher than if only one beet or batch of beets were used. All statistics were performed by the SAS statistical analysis system, and data were analyzed using the general linear model.

RESULTS AND DISCUSSION

Results are summarized in Table 3.2, which lists mean values of microliters of beet juice liberated from the sample during the test period. Confidence intervals ($P \leq 0.05$) are also included.
Table 3.2. Mean values (with confidence intervals) of μL of beet juice liberated from beet cube

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>σ, S/m</th>
<th>Volume of juice (μL)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>conventional</td>
</tr>
<tr>
<td>42</td>
<td>0.17</td>
<td>4.44±0.97&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>4.26±0.35&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>58</td>
<td>0.17</td>
<td>9.85±1.50&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>11.29±1.46&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>72</td>
<td>0.17</td>
<td>24.05±4.26&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>17.14±1.23&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Table 3.2. Mean values (with confidence intervals) of μL of beet juice liberated from beet cube

a,b,c For the same temperature, mean values followed by the same letter are not significantly different (P ≤ 0.05)

There was a statistically significant diffusion enhancement effect between conventional and ohmic treatments at 42° and 58°C, but not at 72°C. This can be partly explained by examining the σ-T profiles of beetroot during conventional and ohmic heating (for further reference, see Halden et al., (1990)). In both the 42° and 58°C cases, the electrical conductivity of beet tissue during ohmic conditions was higher than the electrical conductivity of the same material under conventional conditions. At 72°C, there was no difference in σ between conventional and ohmic cases. Likewise, there was no statistically significant difference between diffusion of beetroot at 72°C. This suggests that diffusion enhancement can be predicted by comparing the electrical conductivities during
conventional and ohmic conditions. The probable reason for higher electrical conductivities during ohmic heating at lower temperatures is increased mobility of constituents.

The applied voltage also influenced the amount of diffusion. In the 42°C and 58°C cases, the lower $\sigma$ carrier fluid ($\sigma = 0.17$ S/m) showed significantly greater differences in diffusion between conventional and ohmic treatments than the higher $\sigma$ carrier fluid ($\sigma = 0.30$ S/m). This is because the higher $\sigma$ carrier fluid requires less voltage to maintain steady-state temperature than the lower $\sigma$ carrier fluid. Applied voltage has been shown to be linearly correlated with the volume of beet dye diffused (Schreier et al., 1993). Because only two carrier fluids were studied for each temperature, it was not possible to determine whether or not diffusion enhancement was a linear function of applied voltage (see Figure 3.3). However, for the 42°C and 58°C cases, increasing the applied voltage increased the diffusion enhancement to the same extent. No significant difference in diffusion or applied voltage was observed at 72°C.

The percent diffusion enhancement is plotted as a function of temperature for each carrier fluid in Figures 3.4 and 3.5.
Figure 3.3. Applied voltage vs. diffusion enhancement
Figure 3.4. Percent diffusion enhancement during ohmic heating
Figure 3.5. Volume of beet dye liberated into solution at the temperatures tested
The highest diffusion enhancement, 186%, occurred at 42°C with the $\sigma=0.17$ S/m carrier fluid; 108% enhancement was observed at the same temperature with the $\sigma=0.3$ S/m carrier fluid. These values are significantly higher than the 40% enhancement factor obtained by Schreier et al. (1993). The 58°C diffusion enhancement was also significantly higher, 168% and 120% for the low and high $\sigma$ carrier fluids respectively. There was no statistically significant diffusion enhancement of the ohmic case at the 72°C data point.

At around 60°C, the cellular structure of beet begins to break down, and significant amounts of beet dye are released into solution. For the conventional heating case, some diffusion of beet dye occurs below this temperature, but large amounts of beet dye are released after this temperature is reached. Schreier's prediction of a 40% diffusion enhancement was integrated over a range of temperatures. At temperatures below the 60°C, the diffusion enhancement seen in ohmic heating is higher than Schreier's estimate. Above 60°C, the extent of diffusion for both treatments approaches the same value.

Our study lends itself to estimates of the extent of diffusion during holding periods of food processing. This information could be used to optimize the extraction of desirable components at specific temperatures. Because of the heightened diffusion effects at lower temperatures during ohmic heating, heat labile components could be effectively isolated. These results also suggest that electric field strength will affect the extent of diffusion during ohmic heating. This could be beneficial in extraction processes, as the addition of an electric field may increase the efficiency of extraction. However, during processes such as sterilization, the leaching of essential nutrients from foods during ohmic heating is also
possible. Preliminary studies in our lab showed that approximately 15% of the ascorbic acid in turnip diffused into carrier fluid after five minutes of ohmic heating. This could result in decreased vitamin consumption if the carrier fluid is not utilized by the consumer.
CONCLUSIONS

The amount of beet dye diffusing into solution was significantly enhanced during ohmic heating at 42°C and 58°C but not at 72°C. These differences were directly proportional to the differences in beet tissue $\sigma$ during conventional and ohmic heating. Applied voltage was positively correlated with diffusion enhancement.
### NOTATION

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>alternating current</td>
</tr>
<tr>
<td>P</td>
<td>probability</td>
</tr>
<tr>
<td>RMS</td>
<td>root mean square</td>
</tr>
<tr>
<td>rpm</td>
<td>revolutions per minute</td>
</tr>
<tr>
<td>σ</td>
<td>electrical conductivity, S/m</td>
</tr>
<tr>
<td>T</td>
<td>temperature, °C</td>
</tr>
<tr>
<td>t</td>
<td>time, seconds</td>
</tr>
</tbody>
</table>
REFERENCES


CHAPTER 4

THE EFFECT OF FREQUENCY AND WAVEFORM ON THE ELECTRICAL CONDUCTIVITY-TEMPERATURE PROFILES OF TURNIP TISSUE

ABSTRACT

Electrical conductivity (σ)-temperature (T) curves were determined as a function of frequency and wave form of alternating current. Turnip cylinders were sandwiched between titanium electrodes and heated ohmically to 100°C using four frequencies (4, 10, 25 and 60 Hz.) and three wave forms (square, sine and sawtooth). The σ-T curve shifted upward at 4 Hz for all wave forms. Curves were not significantly different at 10, 25 and 60 Hz. for sawtooth and sine wave forms, while those for square waves were not significantly different at any of the frequencies tested. A comparison of wave forms showed that σ-T curves for square waves were shifted downward significantly with respect to sine and sawtooth waves. Heating rate increased with decreasing frequency.
INTRODUCTION

The electrical conductivity of a food product is a critical parameter affecting ohmic heating of foods, as it has been shown to influence both heat and mass transfer properties. Studies have been conducted on the changes of electrical conductivities of foods during ohmic heating. Halden et al. (1990) found that the electrical conductivity changes significantly as food properties change during ohmic heating at 50 Hz. Sastry and Palaniappan (1991a,b) modeled the electrical conductivity of various food samples and found that σ increased linearly with temperature at sufficiently high electric field strengths. These investigators also modeled the electrical conductivity of liquid-particle mixtures (1991c). Wang and Sastry (1993) determined that salt diffusion into vegetable tissue was an effective pretreatment for ohmic heating because it raised the electrical conductivity of the tissue. In Chapter 3, it was demonstrated that the electrical conductivity of a beet sample at specific temperatures was an indicator of the extent of mass transfer of beet dye. Although studies concerning electrical conductivity have been numerous and varied, very little has been done to examine the electrical conductivity of foods at low frequencies of alternating current.

Most ohmic heating research to date has been done at frequencies of 60 Hz. (standard frequency for AC current in the United States) or 50 Hz. (standard frequency for AC current in the United Kingdom). Some researchers have investigated the effect of frequency on ohmic heating of foods. Imai et al. (1995) studied the ohmic heating of
Japanese white radish as a function of frequency from 50 to 10,000 Hz. at E=40 V/cm. They found that as frequency increased, the time for the radish to reach the final temperature of 80°C increased. The wave form of alternating current is also of interest. Kim and Pyun (1995) examined the diffusion of soluble components from soybean during ohmic heating as a function of frequency (500 - 10,000 Hz.) and wave form of alternating current (sine waves and square waves). They found that the best soy milk yield occurred at 1,000 Hz., and that the sine wave was more suitable for the extraction of soy milk than the square wave at this frequency. Altering the frequency and wave form of alternating current during ohmic heating has been shown to influence the heat and mass transfer properties of foods.

The objective of this study was to determine the effect of frequency and wave form on electrical conductivity-temperature relationships and the heating rates of ohmically heated turnip.

MATERIALS AND METHODS

Turnip cylinders 2.35 cm. in diameter and 1.3 cm. thick were loaded into the apparatus (described below), and the electrodes sandwiched at the ends of the sample. Considerable care was taken to maintain full contact between the sample and the electrodes, so a uniform heating rate was assumed. Four frequencies (4, 10, 25 and 60 Hz.) and three wave forms (square, sine and sawtooth) were used to heat the samples from
25° to 100°C. A datalogger program was used to monitor time, temperature, current and voltage at two second intervals. Electrical conductivities were determined using the method described by Palaniappan and Sastry (1991a):

\[ \sigma = \frac{L}{AR} \]  

The turnip sample was subjected to a 30.8 V/cm electric field for ten of the twelve frequency-wave form combinations. The two exceptions were the 4 Hz. sine and 4 Hz. sawtooth wave form combinations, in which the maximum voltage obtained by the power supply was used. This corresponded to an electrical field strength of 15.4 V/cm. RMS voltage was used to eliminate electric field strength as a variable when comparing waveforms.

_Equipment details._ The apparatus consisted of a polycarbonate cylinder drilled with a 2.35 cm. diameter hole, the ends of which were fitted with two coated titanium electrodes (coating supplied by APV Company, Devon, England). A hole equipped with a pressurized fitting was drilled into the cylinder for a teflon coated type T thermocouple to monitor center temperature. Electrode seals were maintained with o-rings. The apparatus was designed to minimize evaporation of the sample.

A square wave generator (see Appendix) was used to subject the sample to square waves, while a variable frequency power supply (Elgar Corporation, San Diego, CA) was
used to generate sine and sawtooth waves. Six to eight repetitions of $\sigma$-$T$ curves were performed, and statistical significance was determined using Student's t-test ($P \leq 0.05$).

RESULTS AND DISCUSSION

*Effect of frequency on $\sigma$-$T$ profiles.* Electrical conductivity-temperature curves for the three different waveforms as a function of frequency are presented in Figures 4.1-4.3. The data with statistical details are in Tables 4.1-4.3.

![Graph of Electrical conductivity vs. Temperature]

**Figure 4.1.** Mean $\sigma$-$T$ curves for the sine wave as a function of frequency
Table 4.1. Mean values of $\sigma \pm 1$ standard deviation at selected temperatures for sine waves

<table>
<thead>
<tr>
<th>Frequency, Hz</th>
<th>Temperature, °C</th>
<th>30</th>
<th>50</th>
<th>75</th>
<th>95</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>$0.433\pm0.052^a$</td>
<td>$0.794\pm0.169^a$</td>
<td>$1.31\pm0.228^a$</td>
<td>$1.70\pm0.217^a$</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>$0.107\pm0.039^b$</td>
<td>$0.406\pm0.067^b$</td>
<td>$0.778\pm0.119^b$</td>
<td>$0.961\pm0.114^b$</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>$0.059\pm0.017^c$</td>
<td>$0.404\pm0.137^c$</td>
<td>$0.956\pm0.237^c$</td>
<td>$1.22\pm0.242^c$</td>
<td></td>
</tr>
<tr>
<td>60</td>
<td>$0.033\pm0.004^ex$</td>
<td>$0.318\pm0.164^ex$</td>
<td>$0.758\pm0.249^ex$</td>
<td>$0.967\pm0.243^ex$</td>
<td></td>
</tr>
</tbody>
</table>

For the same temperature, values followed by the same letter are not statistically different (two-tailed pooled t-test, $P \leq 0.05$)

For the same frequency and temperature, values followed by the same letter or are statistically different (two-tailed pooled t-test, $P \leq 0.05$)
Figure 4.2. Mean $\sigma$-$T$ curves for the sawtooth wave as a function of frequency
Table 4.2. Mean values of $\sigma \pm 1$ standard deviation at selected temperatures for sawtooth waves

For the same temperature, values followed by the same letter are not statistically different (two-tailed pooled t-test, P ≤ 0.05)

For the same frequency and temperature, values followed by the same letter or are statistically different (two-tailed pooled t-test, P ≤ 0.05)
Figure 4.3. Mean $\sigma$-T curves for square wave as a function of frequency
Table 4.3. Mean values of ±1 standard deviation at selected temperatures for square waves

<table>
<thead>
<tr>
<th>Frequency, Hz</th>
<th>Temperature, °C</th>
<th>30</th>
<th>50</th>
<th>75</th>
<th>95</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td></td>
<td>0.048±0.023&lt;sup&gt;x&lt;/sup&gt;</td>
<td>0.233±0.140&lt;sup&gt;yx&lt;/sup&gt;</td>
<td>0.554±0.224&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.769±0.243&lt;sup&gt;xv&lt;/sup&gt;</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>0.042±0.014&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.167±0.060&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.432±0.070&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.646±0.033&lt;sup&gt;xv&lt;/sup&gt;</td>
</tr>
<tr>
<td>25</td>
<td></td>
<td>0.038±0.011&lt;sup&gt;xz&lt;/sup&gt;</td>
<td>0.257±0.162&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.583±0.170&lt;sup&gt;xv&lt;/sup&gt;</td>
<td>0.789±0.130&lt;sup&gt;xv&lt;/sup&gt;</td>
</tr>
<tr>
<td>60</td>
<td></td>
<td>0.042±0.017&lt;sup&gt;xz&lt;/sup&gt;</td>
<td>0.217±0.065&lt;sup&gt;xz&lt;/sup&gt;</td>
<td>0.603±0.148&lt;sup&gt;xz&lt;/sup&gt;</td>
<td>0.853±0.155&lt;sup&gt;xz&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Table 4.3. Mean values of σ±1 standard deviation at selected temperatures for square waves

- **a,b,c** For the same temperature, values followed by the same letter are not statistically different (two-tailed pooled t-test, P≤0.05)

- **x,y,z** For the same frequency and temperature, values followed by the same letter or are statistically different (two-tailed pooled t-test, P≤0.05)
For the sine and sawtooth shaped waveforms, the curves at 4 Hz. were significantly different from those at 10, 25 and 60 Hz. Although the 4 Hz. $\sigma$-T curve for the square wave case was also shifted up from the other frequencies, the difference between it and the other curves was not statistically significant. For the sine and sawtooth wave forms, the electrical conductivity-temperature curves at 10, 25 and 60 Hz. were not different from each other. There was no significant effect of frequency for square waves; a slight upward shift at 4 Hz. was observed.

Effect of wave form on $\sigma$-T profiles. Figures 4.4-4.7 show $\sigma$-T profiles at the four frequencies tested for the three wave forms. Tables 4.1-4.3 show corresponding statistical information.

![Graph showing $\sigma$-T profiles](image)

Figure 4.4. $\sigma$-T profiles as a function of wave form at 4 Hz.
Figure 4.5. $\sigma$-$T$ profiles as a function of wave form at 10 Hz.
Figure 4.6. $\sigma$-T profiles as a function of wave form at 25 Hz.
Figure 4.7. $\sigma$-T profiles as a function of wave form at 60 Hz.
The electrical conductivity of the square wave form was lower than the sine and sawtooth wave forms at all frequencies. This downward shift was statistically significant at 4, 10 and 25 Hz. but not at 60 Hz. The electrical conductivity is influenced by changing the wave form, and this influence seems to increase as the frequency decreases. A downward shift in electrical conductivity results in a slower heating rate and could result in reduced mass transfer. Kim and Pyun (1995) found that the square wave was less efficient than the sine wave during ohmic heating for the extraction of soy milk from soybeans. They observed that the square wave caused changes in the dissociation of chemical constituents, as the pH of the extraction process was between 7 and 8 for the square wave, and between 6 and 7 for the sine wave.

*Effect of frequency and wave form on heating rate.* The higher the frequency, the slower the heating rate, as shown in the form of time to reach 100°C, in Table 4.4.

<table>
<thead>
<tr>
<th>frequency, Hz.</th>
<th>wave form</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>sawtooth</td>
<td>sine</td>
</tr>
<tr>
<td>4</td>
<td>183*</td>
<td>157*</td>
</tr>
<tr>
<td>10</td>
<td>120</td>
<td>120</td>
</tr>
<tr>
<td>25</td>
<td>212</td>
<td>326</td>
</tr>
<tr>
<td>60</td>
<td>393</td>
<td>441</td>
</tr>
</tbody>
</table>

Table 4.4. Time (seconds) for turnip center to reach 100°C at E=30.8 V/cm (*E=15.4 V/cm)
Assuming that the resistance component of impedance is largely responsible for the heating, one could expect that at 30.8 V/cm, the times for the triangular and sine wave forms would be approximately one quarter the values measured at 15.4 V/cm.

A graphic representation of this phenomenon can be seen in Figure 4.8. While only triangular waves are included here, the effect is analogous for sine waves.

![Figure 4.8. Time-temperature profiles as a function of frequency for sawtooth waves](image)
For all frequencies except 4 Hz., a very slow increase in temperature occurred until a critical time, after which the temperature increased dramatically. The heating rate after this critical time was the same for all frequencies and occurred at approximately 60°C; thus, it is assumed that the same phenomena are responsible for the temperature increase. At 4 Hz., although the initial heating rate was similar to the other frequencies, a different mechanism clearly occurred, as there was no slow increase in temperature before the sharp increase, and a decrease in the heating rate at 55°C such that the sample approached 100°C relatively slowly.

The square wave time-temperature data showed similarities with the other wave forms. The time for the center of the turnip to reach 100°C increased as the frequency increased, and a dramatic increase in temperature followed a slow temperature increase. However, the 4 Hz. square wave had a time-temperature curve that was similar in shape to the square wave curves generated at the other three frequencies. The decrease in heating rate observed at approximately 55°C with the 4 Hz. sine and sawtooth waves did not occur with the square wave.

The results obtained for time-temperature plots are similar to those obtained by Imai et al., who found that as frequency was increased from 50 to 10,000 Hz., the time required for a Japanese radish sample center to reach 80°C increased approximately sixfold. These investigators found the same heating rate occurred above 60°C and was independent of frequency, but also found that the higher the frequency, the longer the critical time before the heating rate increased. They concluded that the lower frequencies
of ohmic heating damaged the cell membranes because pressurizing the radish to damage the cell membranes also resulted in the immediate fast heating rate. Electroporation was thought to be responsible for this effect.

In this study, the dramatic temperature increase occurred immediately at 4 Hz. for sine and sawtooth waves, and with a minimal delay for a square wave, possibly indicating a change in cellular structure, which resulted in a decreased resistance to current flow and increased moisture transport.

Large variations in $\sigma$-T and time-temperature curves were obtained. Figure 4.9 illustrates six $\sigma$-T curves generated under identical conditions. The curves appeared to follow two distinct patterns, and structural variations were thought to cause these differences. Turnip samples were bored perpendicular to vascular tissue, and electrodes were sandwiched perpendicularly as well. Structural variations exist throughout the cross section of turnip due to the distribution of vascular tissue (Duckworth, 1966). The specific location of the bore and corresponding structural differences were not considered during experimentation. Upon examining the data, we believe that the effect of structure on the electrical conductivity of foods non-homogenous in nature is worthy of additional study.
Figure 4.9. Six trials of an $\sigma$-$T$ profile for 60 Hz sine wave
Electroosmosis has been proposed by Halden et al. (1990) and Schreier et al. (1993) respectively to explain the increase in electrical conductivity and dye diffusion observed when comparing ohmic heating with conventional heating. Experiments conducted by Kim and Pyun (1995) and Imai et al. (1995) suggest that the frequency of alternating current affects the efficiency of extraction and the heating rate. We have demonstrated that the frequency and waveform of applied voltage affect the electrical conductivity. Taken collectively, these studies suggest that a frequency dependent mechanism, possibly electroosmosis, is responsible for these effects seen during ohmic heating. Further research is necessary to characterize the specific mechanisms that govern ohmic heating.
CONCLUSIONS

The electrical conductivity of turnip was found to be a function of the frequency and wave form of applied voltage. The electrical conductivity was significantly higher for sine and sawtooth waves at 4 Hz., but not at 10, 25 and 60 Hz. No significant difference in electrical conductivity was observed from 4-60 Hz. using the square wave, although an upward shift was observed at 4 Hz. Electrical conductivities for square waves were lower than sine and sawtooth waves. This trend was statistically significant at 4, 10 and 25 Hz., but not at 60 Hz. The heating rate increased as frequency decreased. These data suggest that the influence of frequency and wave form on electrical conductivity is increasingly pronounced as frequency decreases.
### NOTATION

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>cross sectional area of sample, cm²</td>
</tr>
<tr>
<td>AC</td>
<td>alternating current</td>
</tr>
<tr>
<td>E</td>
<td>electric field strength, V/cm</td>
</tr>
<tr>
<td>$E_{cr}$</td>
<td>critical electromotive force, V</td>
</tr>
<tr>
<td>f</td>
<td>frequency, Hz.</td>
</tr>
<tr>
<td>L</td>
<td>length of sample, cm.</td>
</tr>
<tr>
<td>R</td>
<td>resistance, ohms</td>
</tr>
<tr>
<td>RMS</td>
<td>root mean square</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>electrical conductivity, S/m</td>
</tr>
<tr>
<td>t</td>
<td>time, seconds</td>
</tr>
<tr>
<td>T</td>
<td>temperature, °C</td>
</tr>
<tr>
<td>V</td>
<td>volts, V</td>
</tr>
</tbody>
</table>
REFERENCES


Lima, M. 1996. Diffusion of beet dye during electrical and conventional heating at steady-state temperature. Doctoral dissertation, the Ohio State University, Columbus, OH.


CHAPTER 5

THE EFFECTS OF OHMIC HEATING FREQUENCY ON HOT-AIR DRYING RATE, DESORPTION ISOTHERMS AND JUICE YIELD

ABSTRACT

Ohmically pretreating vegetable samples has been shown to increase hot-air drying rate, shift desorption isotherms, and improve juice yields with respect to samples pretreated with conventional, microwave and no heating. The frequency of alternating current applied to foods being ohmically heated has been found to affect heat and mass transfer properties. In this study, the hot-air drying rate, desorption isotherms and rehydration behavior of yam, and the juice yields of apples, were compared using a 60 Hz. sine wave and a 4 Hz. sawtooth wave to determine if lowering the frequency could result in additional improvements to these processes. The 4 Hz. sawtooth wave resulted in a faster hot-air drying rate of yam cylinders than the 60 Hz. sine wave. The electric field strength did not affect the drying curves in the range tested at 60 Hz., but did affect the drying rate at 4 Hz. Shifts were not observed in desorption isotherms in higher relative
humidity environments due to mold growth. Ohmically pretreated yam samples maintained their color better than raw samples, and regained their initial shape and color when rehydrated. Juice yield was greatest at the lowest frequency tested. The pretreatment time at 4 Hz. was drastically less than the pretreatment 60 Hz.; both pretreatments softened the apple tissue, reducing input work for the extraction.

INTRODUCTION

Ohmic heating has been shown to influence mass transfer properties. Halden et al. (1990) observed an increase in beet dye diffusion during ohmic heating. This work was extended by Schreier et al. (1993), who demonstrated that the diffusion enhancement in the ohmic case is a linear function of RMS electric field and directly proportional to surface area of the particle; these investigators propose electro-osmosis as the mechanism for diffusion enhancement. In Chapter 3, it was shown that diffusion from a beet sample depends on the electrical conductivity of the beet, and that differences in $\sigma$ between conventional and ohmic conditions account for the difference in the extent of diffusion.

Several other studies have demonstrated the effect of frequency and wave shape of alternating current on the ohmic heating of foods. The ohmic heating of turnip as a function of frequency and wave shape of alternating current was investigated in Chapter 4. Electrical conductivity-temperature ($\sigma$-T) profiles were shifted upward at 4 Hz. but not at 10, 25 and 60 Hz. Sine and sawtooth shaped waveforms gave similar profiles for all
cases except 4 Hz., during which sawtooth waves had significantly higher electrical conductivities between 50° and 65°C. The lower the frequency, the faster the sample reached elevated temperatures. Imai et al. studied the ohmic heating of Japanese white radish from 50 to 10,000 Hz. They found that 50 Hz. gave the sharpest initial temperature increase and took the shortest time to reach 80°C, and theorized that electroporation of the tissue membrane was responsible for this effect. H-NMR analysis of ohmically heated tissue showed more free movement of liquid components than raw samples.

There has been some effort to use the attributes specific to ohmic heating to improve various aspects of food and industrial processing. Electric fields were used to increase the efficiency of sucrose extraction from sugar beet (Katrokha et al., 1984). Kim and Pyun (1995) used ohmic heating to enhance the diffusion of soy milk from soybeans as a function of frequency and wave form. Solid and protein yields were enhanced approximately 16% and 25% respectively when the soy slurry was heated using a 12.5 V/cm voltage gradient. Carlon and Latham (1992) studied the enhanced drying rates of wetted materials in electric fields, and found that the drying time of wetted paper towel discs decreased by a factor of 6 when the electric field strength was increased from 0 to 7,000 V/cm.

Wang (1995) compared raw samples with those pretreated using conventional, microwave and ohmic heating at 60 Hz., and found that ohmic heating resulted in the largest enhancement of the drying rate of carrot, yam and potato cylinders. Desorption isotherms of these samples were shifted at higher water activities such that at a constant
equilibrium moisture content, the water activity of ohmically pretreated samples was higher than untreated, conventionally treated and microwave treated samples. Wang theorized that these differences could be attributed to the diffusion of intracellular moisture to the intercellular space, and suggested that since ohmic pretreatment (done at 60 Hz.) results in significantly better drying rates than microwave pretreatment (2450 Hz.), frequency may play an important role in moisture diffusion. Ohmic heating was also found to enhance the extraction of apple juice from Red Delicious apples.

Lowering the frequency of alternating current during ohmic heating may result in improved mass transfer effects. The basis for this hypothesis is drawn from the previous literature and ongoing work, and, as shown in Chapter 4, the highest electrical conductivity was obtained using a 4 Hz. sawtooth wave. It may be worthwhile to study the influence of frequency of pretreatment on the efficiency of mass transfer operations.

Accordingly, the objective of this study is to compare ohmic pretreatments to determine if additional improvements to drying, sorption, rehydration and extraction are obtained.

**MATERIALS AND METHODS**

Ohmic pretreatment consisted of sandwiching the food sample, 2.35 cm. in diameter and 1 cm. thick, between two titanium coated electrodes, and heating the sample ohmically until the geometric center of the sample reached a given temperature (specific
details follow). A variable frequency power supply (Elgar Corporation, San Diego, CA) was used to generate alternating current for heating the sample; two configurations were used, a 4 Hz. sawtooth wave, and a 60 Hz. sine wave. The 4 Hz. sawtooth waveform had shown far higher electrical conductivities than other waveforms and frequencies in earlier studies (see Chapter 4). A Teflon-coated thermocouple was inserted into the sample's geometric center to monitor temperature. Raw (untreated) samples were also drilled with the thermocouple in order to ensure that all samples were comparable. Samples were pretreated immediately after being cut to the proper dimensions. The aforementioned methods were used in all experiments unless otherwise noted.

*Hot-air drying curves.* Alternating current at various voltage gradients was used to pretreat the sample; for the 4 Hz. sawtooth wave, 20 and 40 V/cm gradients were used, while the 60 Hz. sine wave was subjected to 40, 60 and 70 V/cm field strengths. Accurate control of endpoint temperature was possible with all electric field strengths except 70 V/cm, thus results under this condition should be interpreted with care. To obtain a voltage gradient of 40 V/cm for the 4 Hz. case, the sample thickness was halved to 0.5 cm. To ensure valid comparison, a half sample thickness was also used for the control and 60 Hz. sine wave pretreatments at this same voltage gradient.

After the sample reached 80°C, the power was immediately turned off and the pretreated sample placed in an aluminum dish, weighed and loaded into a food dehydrator (Excalibur Products, Sacramento, CA) for hot-air drying. The dehydrator was maintained at a temperature of 51.7°C, and the relative humidity was 10%, measured with a dew
point sensor. Moisture content data were obtained by periodic weighing of the samples with a balance. Samples in the dehydrator were located at the center line, which was perpendicular to the direction of the hot air, to avoid position variation. After 6.5 hours of dehydration the sample was transferred to a 120°C oven for 24 hr. to determine moisture content. Raw samples with no pretreatment were dehydrated in the same way for reference. All samples were tested in three replications, and Student's t-test ($P \leq 0.05$) was used for hypothesis testing.

*Desorption isotherms.* Samples pretreated in the method described above were cut into equal portions (each approximately 0.4 g), then placed into aluminum pans. The samples and pans were loaded into desiccators maintained at different relative humidities (details below), and the weight monitored daily. Moisture equilibrium was assumed when the weight difference between two successive days was less than 0.001 g. This took four to nine days. After reaching equilibrium the moisture content of the samples was determined by drying the samples in a 120°C oven for 24 hrs. Three repetitions of each pretreatment and raw sample were tested, and Student's t-test ($P \leq 0.05$) was used for statistical comparison.

Relative humidity control was achieved with saturated salt solutions, which were prepared corresponding to a range of relative humidities of 0.11 to 0.98, and transferred to desiccators. Table 5.1 shows the chemicals used in the experiment and the literature values of the relative humidity of the saturated solutions. To avoid moisture exchange
between desiccators and the ambient environment, vacuum grease was used to ensure sealing. After setup the desiccators were left for several days to reach equilibrium.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Relative humidity, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>LiCl</td>
<td>11</td>
</tr>
<tr>
<td>KC$_2$H$_3$O$_2$</td>
<td>23</td>
</tr>
<tr>
<td>MgCl$_2$</td>
<td>33</td>
</tr>
<tr>
<td>K$_2$CO$_3$</td>
<td>43</td>
</tr>
<tr>
<td>NaNO$_2$</td>
<td>65</td>
</tr>
<tr>
<td>NaCl</td>
<td>75</td>
</tr>
<tr>
<td>KCl</td>
<td>86</td>
</tr>
<tr>
<td>CaSO$_4$</td>
<td>98</td>
</tr>
</tbody>
</table>

Table 5.1. Literature values of relative humidity of saturated solutions (Rockland, 1960 and Labuza et al., 1985)

*Rehydration studies.* Yam samples were pretreated using the same procedure for drying and sorption studies. Immediately following pretreatment, samples were cut into approximately 0.4 g portions, put into aluminum dishes and placed in 43% and 75% relative humidity desiccators for approximately three weeks to simulate storage conditions. Subsequently, the yams were rehydrated by filling the aluminum dishes with water at room temperature and waiting 35 minutes. Photographs and visual observations were conducted before and after rehydration.
Juice extractions. The apparatus used was the same as Wang (1995) and consisted of an extraction chamber, plunger fitted with an O-ring at the lower end and an Instron Universal Testing Machine (IUTM). The plunger was attached to the cross head of the IUTM, which provided the motion mechanism. The base of the extraction chamber was detachable for unloading samples and cleaning. Two openings were located on opposite sides of the base; air pressure was supplied at the upper opening during extraction to facilitate juice extraction at the lower one.

The samples were Red Delicious apples, which were purchased from the same source and used the same day for experiments; the temperature of the apples was ambient at all times. To prepare samples for juice extraction, four apples were cut in half and separated into two groups, with each of the four apples having a half in each group. Halves were covered with parafilm to minimize exposure to air. One sample was cut from each half. These four cylinders were subjected to pretreatment, weighed, stacked on top of each other and wrapped in a nonabsorbent nylon cloth, then transferred to the chamber for juice extraction. The four apple halves in each group were used to make three extractions, one for each pretreatment (raw apple, pretreatment with 4 Hz. sawtooth wave, pretreatment with 60 Hz. sawtooth wave). In this way, sample deviations could be eliminated as a variable.

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1This instance involved a 60 Hz. sawtooth wave, which had no statistically significant difference from the 60 Hz. sine wave (see Chapter 4 for details).
Ohmic heating was conducted by sandwiching each apple sample between two titanium coated electrodes and heating to 40°C by electricity. To accurately control endpoint temperature, a 40 V/cm voltage gradient was used for heating.

The extraction was performed by pressing the sample stack from its original height of 4 cm. to 0.5 cm. The plunger was pressed at a rate of 3 cm/min. Apple juice was collected in a small beaker, and volume was measured using a set of pipettes accurate to 10 μL. Juice yield was determined by dividing the volume of juice collected (mL) by the mass of the sample (kg). Seven replications of each treatment were made, and statistical significance was determined using Student's t-test (P ≤ 0.05).

RESULTS AND DISCUSSION

Drying curves. Mean dimensionless drying ratio vs. time for the three treatments is shown in Figure 5.1. The 4 Hz. sawtooth pretreatment yielded the fastest drying rate at all times. Data are in Table 5.2.
Figure 5.1 Drying curves of raw and ohmically pretreated yam cylinders (E=40 V/cm)

Table 5.2 Data on mean dimensionless moisture ratio of 0.5 cm thick yam cylinders after 2, 4 and 6 hr. of hot-air drying; ± 1 standard deviation reported

<table>
<thead>
<tr>
<th>Pretreatment</th>
<th>1 hr. drying</th>
<th>2 hr. drying</th>
<th>4 hr. drying</th>
<th>6 hr. drying</th>
</tr>
</thead>
<tbody>
<tr>
<td>raw</td>
<td>0.6722±0.0133a</td>
<td>0.4526±0.0121a</td>
<td>0.2024±0.0006a</td>
<td>0.0348±0.0016a</td>
</tr>
<tr>
<td>60 Hz. sine</td>
<td>0.6605±0.0138a</td>
<td>0.4397±0.0133a</td>
<td>0.1778±0.0032b</td>
<td>0.0253±0.0065ab</td>
</tr>
<tr>
<td>4 Hz. sawtooth</td>
<td>0.6307±0.0108a</td>
<td>0.4146±0.0130b</td>
<td>0.1567±0.0049c</td>
<td>0.0225±0.0020b</td>
</tr>
</tbody>
</table>

a, b, c for the same drying time, mean values followed by the same letter are not significantly different (two tailed pooled t-test, P≤0.05)
Although a statistically significant difference was achieved between the 4 Hz. sawtooth pretreatment and no pretreatment at all times, these data are not as pronounced as those obtained by Wang (1995). The 60 Hz. sine wave pretreatment in this work was not significantly better than no pretreatment at some of the drying times. These data show that the 4 Hz. sawtooth pretreatment results in a faster drying rate than the 60 Hz. sine wave pretreatment, indicating that changing the frequency and waveform of alternating current affects rate of drying.

Although the 4 Hz. sawtooth pretreatment results in a faster drying rate at all times, the increased drying rate effect is most pronounced for intermediate moisture contents. This suggests that ohmic pretreatment is most advantageous when drying foods to intermediate moisture contents, for example dried fruits and nuts, and some types of candy. Some data in the Wang (1995) study show similar trends, with pretreatment effects being less pronounced as the moisture content drops. Drying rates of pretreated samples approach those of untreated samples as they progress through the falling rate period(s) of drying, suggesting that internal resistance to moisture transfer at low moisture contents is comparable, regardless of pretreatment.

The ohmically pretreated samples showed visible surface moisture, while the untreated samples did not. Surface moisture was evident almost immediately with the 4 Hz. pretreatment, with a very small increase in temperature (<5°C). It took slightly longer for surface moisture to become evident during 60 Hz. ohmic heating. These observations are indicative of a migration of moisture that appears to be occurring with ohmic heating.
The results and observations noted here support the hypothesis that decreasing the frequency of alternating current during ohmic heating results in increased mass transfer effects.

The effects of electric field strength on the drying rate are shown in Figures 5.2 and 5.3, which are curves for the 4 Hz. sawtooth pretreatment at electric field strengths of 20 and 40 V/cm, and for the 60 Hz. sine wave pretreatment at 40, 60 and 70 V/cm.

The 40 V/cm electric field resulted in a faster drying rate than the 20 V/cm field strength for the 4 Hz. pretreatment case. No difference in drying was obtained for the 40 and 60 V/cm pretreatments at 60 Hz. The drying rates obtained using the 4 Hz. pretreatment seem to be more sensitive to changes in the electrical field strength than the 60 Hz. pretreatment.

The drying curve for the 60 Hz. sine wave and the 70 V/cm electric field strength was not as efficient as the 40 and 60 V/cm fields. This result seemed unexpected, thus, three more repetitions were performed, which yielded the same results. At 70 V/cm the endpoint temperature was impossible to control; as the temperature approached 80°C the voltage was switched off, but the final temperature at the geometric center of the yam reached 95°-105°C in most cases. These temperatures are sufficiently high to cause increased gelatinization of starch in the yam sample, which results in a hindrance of moisture transfer (Luck, 1981, see later discussion). This is may be the reason for the decreased drying rate at the higher field strength.
This result could be important when designing processes for drying foods using this technology. While increasing the electric field strength will probably increase the drying rate, the temperatures obtained in the food product during ohmic heating can change its physical properties.

Figure 5.2. Effect of electric field strength on drying curves for the 4 Hz. ohmic pretreatment
Figure 5.3. Effect of electric field strength on drying curves for the 60 Hz. ohmic pretreatment
Desorption isotherms. Desorption isotherms are shown in Figure 5.4. Data are in Table 5.3.

Figure 5.4 Desorption isotherm of yam samples (raw and pretreated to 80°C)
<table>
<thead>
<tr>
<th>$a_w$</th>
<th>raw</th>
<th>4 Hz. sawtooth</th>
<th>60 Hz. sine</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.11</td>
<td>0.251±0.027&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.323±0.026&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.300±0.018&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>0.23</td>
<td>0.274±0.038&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.322±0.028&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.324±0.034&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>0.33</td>
<td>0.276±0.018&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.332±0.026&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.335±0.030&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>0.43</td>
<td>0.271±0.031&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.343±0.025&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.332±0.022&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>0.65</td>
<td>0.376±0.017&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.391±0.036&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.423±0.040&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>0.75</td>
<td>0.488±0.053&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.551±0.027&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.541±0.034&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Table 5.3. Equilibrium moisture contents (db) ± 1 standard deviation

<sup>a,b</sup> For the same water activity, mean values followed by the same letter are not statistically different (two-tailed pooled t-test, $P \leq 0.05$)
There was no significant difference in water activity at constant equilibrium moisture content among the treatments at relative humidities of 65% and higher. Wang (1995) found that no difference among treatments was obtained at the lower water activities, but that at higher water activities (RH≥75%), the sorption isotherms were shifted to the right, indicating a migration of moisture from intracellular regions to intercellular (the raw sample has a higher moisture content for a given relative humidity). This phenomenon was not duplicated in this study. This may occur because yam was used for these isotherms, which gave the smallest shift of the samples studied by Wang. The shift is evident at higher relative humidities, but interestingly, ohmically pretreated samples in this region exhibited mold growth, and could not be analyzed.

To confirm that mold growth on ohmically pretreated samples was not an isolated phenomenon, the isotherm experiment was repeated, however, the electrodes were sterilized with a 5% bleach solution before each experiment to ensure that the mold growth was not a direct result of contact with the electrodes. Mold growth was observed within 48 hours for all ohmically pretreated samples and not for the untreated samples. This supports the hypothesis that the increased water activity of the sample obtained in ohmic heating results in better support of microorganism growth. Thus, if ohmic heating is being used in conjunction with a drying process, care must be taken to dry samples rapidly such that microorganism growth does not occur.

For lower relative humidities, equilibrium moisture contents were significantly higher for pretreated samples than for raw ones. This result was also seen in the Wang
(1995) study, although the elevated moisture contents for pretreated samples were not significantly higher than the raw sample. This trend may be explained by starch gelatinization and the behavior of bound water at low moisture contents. Starch gelatinization occurs during heating, and is characterized by the swelling of amorphous regions and melting of crystalline regions of starch granules. A gel is formed as a result, and water is taken up and bound in this structure. Ohmic pretreatments probably result in some starch gelatinization, as the starch gelatinization temperature range of roots and tubers is 56-70°C (Ensminger et al., 1993). Luck (1981) reported that bound water exhibits a decrease in diffusion coefficient with decreasing moisture content. The increased moisture content of pretreated samples at low relative humidity may result from the increase in bound water caused by starch gelatinization.

Color and shape variations between untreated and ohmically pretreated samples were observed during the equilibration period. Untreated samples tended to keep their shape and simply became smaller when held in the lower relative humidity desiccators (RH ≤ 0.75) while the ohmically pretreated samples shriveled. This indicates structural changes in the pretreated samples. Color changes were also observed; the untreated samples faded in color or became gray over time, while the ohmically pretreated samples maintained their color.

These observations prompted further study. Figures 5.5 and 5.6 represent ohmically pretreated yam samples held for three weeks at 43% and 75% relative humidity respectively. When these samples were rehydrated, the following observations were made.
For both relative humidities tested, the untreated samples floated in water, while the ohmically pretreated samples did not. The ohmic samples may have ruptured cell walls and a less tortuous structure so that water could seep in easily and prevent the sample from floating. Also, the shriveling seen in the ohmic case could result in the yam becoming more dense than the untreated sample.

The ohmically pretreated samples also held their color better both before and after rehydration than the non-treated samples. Upon rehydration, the ohmically pretreated samples looked the same as when they were initially placed into the desiccator; the color and shape of the original samples were essentially regained. The untreated samples exhibited faded color and surface irregularity upon rehydration. Differences between treated and untreated samples upon rehydration were not thought to be a result of the rehydration procedure. The untreated sample floated just below the water surface, and all surfaces of the sample were in contact with water at all times. No difference was observed in the rehydration rate among any of the samples.

These observations could be of considerable importance if applied to foods that are dried and rehydrated before consumption. Another particularly promising application would appear to be the production of intermediate moisture foods, where the maximum drying acceleration range of water activities may be used to advantage. While sensory data are not yet available, the improvements in color and texture observed with ohmic pretreatment could result in a vast improvement in product quality, both in already existing
products, and in new products that may be more feasible to dry and rehydrate with this
technology. Further research in this area is recommended.
Figure 5.5. Yam samples held at 43% relative humidity (a) before and (b) after rehydration.
Figure 5.6. Yam samples held at 75% relative humidity before and after rehydration.
Juice extraction. Data on apple juice yield are contained in Table 5.4, which includes juice yields from apple samples with no pretreatment (raw), the 4 Hz. pretreatment and the 60 Hz. pretreatment. Both ohmic pretreatment resulted in a significantly higher juice yields than the untreated sample, and the 4 Hz. pretreatment resulted in more juice yield than the 60 Hz. pretreatment. Apple juice extracted from all samples showed no visual difference in color or clarity; this suggests that the ohmic pretreatment does not cause browning.

<table>
<thead>
<tr>
<th>pretreatment</th>
<th>raw</th>
<th>60 Hz.</th>
<th>4 Hz.</th>
</tr>
</thead>
<tbody>
<tr>
<td>mean</td>
<td>486.4</td>
<td>586.9</td>
<td>609.4</td>
</tr>
<tr>
<td>standard deviation</td>
<td>30.9</td>
<td>9.0</td>
<td>23.1</td>
</tr>
</tbody>
</table>

Table 5.4. Means and standard deviations of apple juice yield (mL/kg) as a function of pretreatment

These results are very encouraging from a processing standpoint. In the Wang (1995) study, differences in the amount of apple juice extracted per kilogram of apple between microwave and ohmic heating were not evident until 50°C. At 40°C, the pretreatment temperature used here, enhanced diffusion in the lower frequency case is already significant. While force-compression curves were not determined for these extractions, a visual observation of the compression force exhibited by the apparatus at the commencement of juice flow showed that less force was needed to begin the extraction of the pretreated samples as compared to the raw sample. A slight decrease in compression force needed for extraction was observed for the 4 Hz. pretreatment with
respect to the 60 Hz. pretreatment, but this effect was not tested statistically. Wang (1995) showed that both microwave and ohmic pretreatments required less input work than the raw material. Thus, in addition to yielding more juice, the ohmic pretreatment also result in less energy input needed to obtain the juice.

Another excellent attribute of the 4 Hz. case is the decreased time needed for pretreatment. Figure 5.7 shows the mean time-temperature history for apple slices to reach the 40°C endpoint temperature for both the 4 Hz. and 60 Hz. pretreatments using an electric field strength of 40 V/cm. The mean time it takes to reach 40°C is 33 seconds for the 4 Hz. case, while the 60 Hz. case takes 109 seconds (means generated using time-temperature profiles of eight apple slices). Using the low frequency pretreatment cuts the time necessary to achieve this effect to less than a third of the value at 60 Hz.

Processes can be improved by increasing their efficiency, and decreasing the amount of time and energy required to complete them. Ohmic pretreatment at low frequency accomplishes all these objectives for juice extractions. While sensory characteristics have not yet been evaluated, visual observation shows no difference between conventionally extracted and ohmically pretreated juice.
Figure 5.7. Time-center temperature profiles for apple slices from room temperature to 40°C (E=40 V/cm)
CONCLUSIONS

The effect of frequency of alternating current applied to food samples is significant, with increased mass transfer effects occurring as the frequency decreases. The highest drying rates and juice yields were obtained using a 4 Hz. frequency of alternating current. Ohmic pretreatment exhibits several novel effects, including tissue softening and an increase in sample surface moisture at relatively low temperatures, and an increased integrity of color when held at low relative humidities over time. These phenomena could represent important ramifications for processing. Using ohmic heating at low frequencies to enhance the diffusion of specific components appears to be an important method for processes involving mass transfer.
NOTATION

\( a_w \)  
water activity

\( m \)  
mass of sample (kg)

\( M \)  
mass of sample (% db)

\( P \)  
probability

\( \text{RMS} \)  
root mean square

\( t \)  
time (s)

\( T \)  
temperature (°C)

\( V \)  
volume of extracted juice (mL)

\( Y \)  
juice yield (mL/kg apple)

\( \sigma \)  
electrical conductivity (S/m)

Subscripts

\( e \)  
equilibrium

\( o \)  
initial
REFERENCES


Lima, M. 1996. Diffusion of beet dye during electrical and conventional heating at steady-state temperature. Doctoral dissertation, The Ohio State University, Columbus, OH.


Wang, W. 1995. Ohmic heating of foods: physical properties and applications. Doctoral dissertation, The Ohio State University, Columbus, OH.
CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

1. Ascorbic acid degradation kinetics in orange juice in the temperature range of 65°-90°C was unaffected by an electric field strength of 18.2 V/cm.

2. Ascorbic acid degradation followed pseudo first order kinetics for both conventional and ohmic heating treatments, and kinetic constants were in the range of reported literature values.

3. Electrolytic reactions (both gas production and dissolution) appeared to occur when using stainless steel electrodes, but were not evident with specially coated titanium electrodes. No difference in ascorbic acid degradation was observed using either set of electrodes.

4. The volume of beet dye diffusing into solution was significantly enhanced during ohmic heating at 42°C and 58°C but not at 72°C; these differences were directly proportional to the difference in beet tissue σ during conventional and ohmic heating.

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5. Applied voltage was positively correlated with diffusion enhancement.

6. The electrical conductivity of turnip was a function of the frequency and wave form of applied voltage.

7. The electrical conductivity was significantly higher for sine and triangular waves at 4 Hz., but not at 10, 25 and 60 Hz.

8. No significant difference in electrical conductivity was observed from 4-60 Hz. using the square wave, although an upward shift was observed at 4 Hz.

9. Electrical conductivities for square waves were lower than sine and triangular waves.

10. The heating rate increased as frequency decreased.

11. The effect of frequency of alternating current applied to food samples was significant, with increased mass transfer effects occurring as the frequency decreased.

12. The highest drying rates and juice yields were obtained using a 4 Hz. frequency of alternating current.

13. Ohmic pretreatment exhibits several novel effects, including tissue softening and an increase in sample surface moisture at relatively low temperatures, and an increased integrity of color when held at low relative humidities over time.

RECOMMENDATIONS

1. Vitamin degradation kinetics for fat soluble vitamins should be studied to ensure that the relationship between fat melting and vitamin retention is characterized.
2. Conditions resulting in leaching of essential nutrients from foods during ohmic heating due to diffusion enhancement should be determined.

3. Electrochemical changes during ohmic heating need to be further investigated to better understand features specific to ohmic heating, such as electrolysis and frequency response.

4. Further rehydration studies should be conducted to investigate the potential of pretreatment for improved processing. Sensory and other characterization studies are also needed.
Figure A.1. Schematic of square wave generator
LIST OF REFERENCES


Lima, M. 1996. Diffusion of beet dye during electrical and conventional heating at steady-state temperature. Doctoral dissertation, The Ohio State University, Columbus, OH.


Wang, W. 1995. Ohmic heating of foods: physical properties and applications. Doctoral Dissertation, The Ohio State University, Columbus, OH.