Electrical Properties of Fish Mince During Multi-frequency Ohmic Heating

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ABSTRACT

A multi-frequency ohmic heating system with 30 Hz~1 MHz range which could deliver 250 watts was developed for measuring electrical conductivity and absolute dielectric loss of food samples. Pacific whiting surimi paste and stabilized mince in the 20~70°C range were tested at frequencies from 55 Hz to 200 kHz. Sample impedance decreased slightly with frequency. The DC electrical conductivity (σ_{dc}) and absolute dielectric loss (ϵ'') of Pacific whiting surimi paste increased with temperature and salt concentration; σ_{dc} and ϵ'' of the stabilized mince increased with temperature. Empirical models of electrical properties for surimi paste (moisture content 79% and salt at 1, 2 or 3%) and stabilized mince (77% moisture and 0.74% salt) were derived. Electrolytic corrosion diminished with frequency.

Key words: fish mince, dielectric properties, ohmic heating, conductivity

INTRODUCTION

LARGE QUANTITIES OF PACIFIC WHITING (*Merluccius productus*) are utilized in the form of surimi, a washed mince which contains a Cathepsin L protease with maximum activity at 55°C (Seymour et al., 1994). This activity causes a breakdown of myofibrillar proteins and inhibits development of a 3 dimensional gel structure (Niwa, 1992). Research and subsequent industry practice have shown that 1~1.5% of a beef plasma protein (BPP) would ensure maximum surimi gel strength, a major functional property influencing its value (Morrissey et al., 1993; NFI, 1991).

Another product form of Pacific whiting is unwashed mince stabilized in frozen storage with various cryoprotectants (Simpson et al., 1994, 1995). Enzyme concentrations measured in such minces are about 10x those of washed and stabilized samples (An et al., 1994a, b). Thus enzyme inhibition in unwashed mince may require inhibitor concentrations far greater than the 1~1.5% BPP typically used in commercial surimi.

Enzyme inhibitors such as BPP have some disadvantages due to labeling restrictions, cost, or sensory effects. Studies, therefore, have been initiated to thermally inactivate the protease enzyme by very rapid heating.

Ohmic, or electrical resistive heat generated internally due to electrical resistance of the sample occurs rapidly (deAlwis and Fryer, 1992; Palaniappan and Sastry, 1991a). Gel strength of surimi from walleye pollock, white croaker, threadfin bream, sardine, or Pacific whiting has been improved when cylindrical samples were ohmically heated, as compared with those heated in a 90°C water bath (Shiba, 1992; Shiba and Numakura, 1992; Yongsawatdigul et al., 1995b). It was shown that 1.9 cm diameter cylindrical samples heated in a 90°C bath could take up to 6 min. to reach 70°C (Yongsawatdigul et al., 1995b), where the protease would be completely inactivated (Seymour et al., 1994). During 2 min. of this process, the center was within the range 40-60°C, during which the protease hydrolyzed myofibrillar proteins and destroyed gel structure. Protease activity was virtually stopped when similar samples were heated ohmically.

Most ohmic heating systems have used 50-60 Hz alternating current. Research has sought to define temperature- and composition-dependent values of electrical conductivity, the critical factor influencing heat generation rates (Halden et al, 1990; Palaniappan and Sastry, 1991a, b; Shreier et al., 1993; Yongsawatdigul et al., 1995a). One constraint of 60 Hz ohmic heating, however, is that electrolytic reactions can take place at the electrode surface, leading to product burning and corrosion of electrodes made from common food-grade metals (Shiba, 1992). To overcome this, increasing electric signal frequency has been suggested; Uemura et al. (1994) and Reznik (1996) reported reduced electrode corrosion when cooking at alternating current frequencies approaching 10 kHz.

Biological materials act as "lossy media" considering their ability to store and dissipate electrical energy from an applied electromagnetic field (Von Hippel, 1954). These properties result from electrical charging and loss currents generally related to the electrical capacitance and resistance of the material. They are defined by fundamental dielectric properties which can be expressed as direct current (DC) electrical conductivity σ_{dc}(S/m), absolute dielectric constant ϵ'(F/m), and absolute dielectric loss ϵ''(F/m).

Inductive materials also have magnetic characteristics related to their inductance and resistance and are defined in terms of absolute complex permeability μ^*(H/m). However, natural biological materials are noninductive dielectrics, do not couple magnetic energy, and hence have a magnetic permeability near that of free space. Thus magnetic coupling effects may be neglected in biological materials (Mudgett, 1985).

To apply ohmic heating of Pacific whiting minced products in higher frequency ranges, our objective was to characterize the properties: DC electrical conductivity σ_{dc}; absolute dielectric constant ϵ'; and absolute dielectric loss ϵ'', during ohmic heating at various frequencies to 200 kHz.

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MATERIALS & METHODS

Sample preparation

Surimi paste. Washed mince of Pacific whiting was taken from the process line of a local manufacturer and mixed with 4% sucrose, 4% sorbitol (ICI Specialties, New Castle, DE) and 0.3% sodium tripolyphosphate (B.K. Ladenburg Corp., Cresskill, NJ) to make surimi at the OSU Seafood Laboratory (Astoria, OR). No enzyme inhibitors were added. Samples were packaged in ~450g containers, frozen in a -30°C room, then brought to our lab in Corvallis, OR and kept in a -30°C room until the experiments. Two packages of surimi were taken from frozen storage, thawed at room temperature (~23°C) for around 2h., then cut into small pieces (about 3 cm cubes). Three batches of surimi paste representing one moisture content (79% wet basis) and three NaCl concentrations (1, 2 and 3% w/w) were prepared. Due to three washing cycles and dewatering during surimi-making, ionic constituents originally present in the fish were removed with wash water, resulting in a low ion concentration in the surimi (Lin, 1992). The sodium concentration of Pacific whiting surimi without enzyme inhibitors was 0.002% (Chung et al., 1993), negligible considering the amount of

NaCl used (1%~3%). Thus, the effect of added NaCl was studied instead of total ion concentration. About 1 kg of partially thawed surimi was chopped at low speed for 1.5 min in a vertical vacuum cutter (Model UM 5 Universal, Stephan Machinery Co., Columbus, OH). Salt was added and mixed with surimi for another 30s. Ice was then added and mixed for another 1 min to adjust the moisture content to the target value. The sample was further chopped at high speed under a vacuum of 600 mm Hg for 3 min. The paste was maintained below 8°C during chopping. Final moisture content of each batch was checked using an infrared moisture determination balance (A&D Co., Ltd., Tokyo, Model AD-4714A). The surimi paste was discharged by a sausage stuffer into CPVC (chlorinated polyvinyl chloride) tubes (1.9 cm dia ∞ 10 cm long). Slow filling was used to ensure complete filling and to avoid introducing air. The sample holders were iced and stored in a household refrigerator until testing within 2h.

Stabilized mince. Stabilized mince was made from Pacific whiting fillets by grinding through a plate with 3.2 mm dia holes. Cryoprotectants of 4% sucrose and 4% sorbitol (ICI Specialties, New Castle, DE) were added at the OSU Seafood Laboratory (Astoria, OR). The stabilized mince was packaged in ~450g units, frozen in a -30°C room, then brought to our lab in Corvallis and kept in a -30°C room. For each experiment, two packages were taken from frozen storage and thawed at room temperature (~23°C) for around 4h. Moisture content, determined by an infrared moisture balance was found to be 77%. Inherent salt concentration was 0.74% (Lin and Park, 1996). The mince was stuffed into CPVC sample holders and stored, as described for samples of surimi paste.

Test circuit and apparatus

An equivalent circuit model was used to represent the food sample (Fig.1), with overall sample impedance Z_{load} consisting of components C_{fish}, R_{dc}, and R_e. The quantity C_{fish} was electrical capacitance, calculated from the absolute dielectric constant ϵ' as $C_{fish} = \epsilon' A/L$, (where A=cross sectional area of sample, L=sample length). Quantity R_{dc} was electrical resistance, calculated from the DC electrical conductivity σ_{dc} as $R_{dc} = L/(\sigma_{dc}A)$. Quantity R_e was electrical resistance, calculated from the absolute dielectric loss ϵ'' as $R_e = L/(2\pi f \epsilon'' A)$, (where f = applied frequency).

The apparent impedance Z_{load} of the sample model was calculated from standard circuit theory. Coefficients ϵ' and ϵ'' would show their effects only at higher frequency levels; hence at low frequency, the overall sample impedance would reduce to the DC electrical resistance R_{dc}. At high frequency, any phase shift between the power output voltage and the voltage across the sample would indicate an effect of ϵ'. As ϵ'' becomes nonzero at high frequencies, R_e would take on a finite value. Once R_{dc} was determined at low frequency, ϵ'' could then be calculated from the value of Z_{load} using a parallel resistor equation.

The sample holder was constructed as described by Yongsawatdigul et al (1995b). Two electrodes from discs of stainless steel 304 with a thickness of ~4 mm, were pushed against excess sample materials as they were inserted into a 19-mm i.d. CPVC sample tube. As pressure was applied, excess sample material was pushed out through a thermocouple access hole in the wall at the center of the sample holder. An air cylinder and regulator applied an electrode pressure of 150 kPa to ensure an air-free contact between electrode and sample. A T-type thermocouple, covered with a thin Teflon tube to prevent interference from the electrical field, was inserted at the geometric center of the sample through the access hole. During heating, temperature was monitored by a datalogger (model 21X, Campbell Scientific, Inc., Logan, UT).

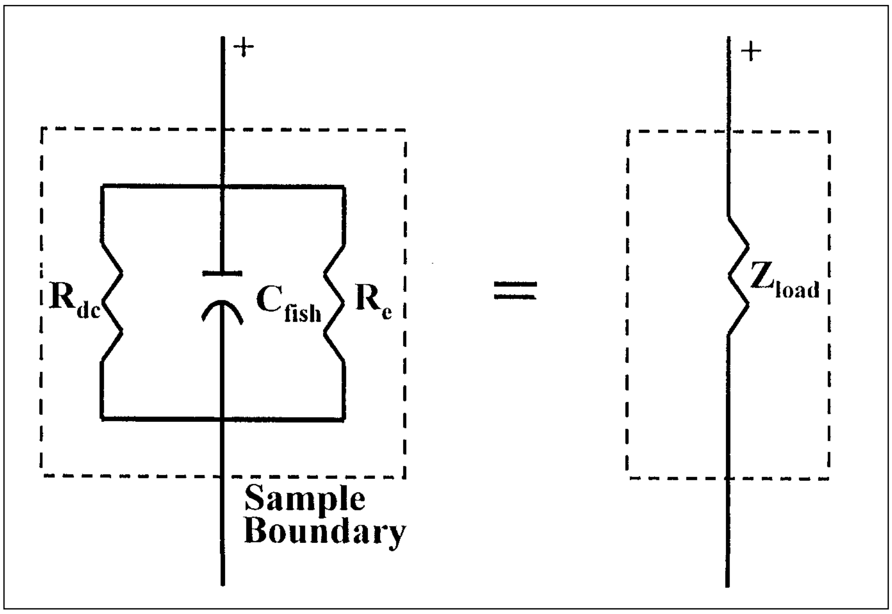

Fig. 1—Equivalent circuit representing food sample.

In the experimental system (Fig. 2), applied frequencies and amplitudes of AC current were set by a Function/Arbitrary Generator (Hewlett Packard, HP 33120A 15 MHz). This enabled generation of various wave forms at frequencies from 0.0001 Hz to 15 MHz at levels from 50 millivolts to 10 volts peak-peak into a 50 ohm load (or about twice that level into a high impedance load). The signal from the function generator was then amplified by a broadband power amplifier (Industrial Test Equipment, Powertron 250A) with an output of about 23V rms when the input was near 1V rms. The amplifier could deliver 250 watts at frequencies to 1 MHz. Amplifier output was sent through a transformer (Industrial Test Equipment, Transformers) to boost voltage to 130V while maintaining the 250 watt power capability. The system used three transformers, selected for low (30 Hz-30 kHz), medium (10 KHz-200 kHz) and high (100 KHz-2 MHz) frequency ranges.

A four channel oscilloscope (Hewlett Packard, HP 54601B) was used to display measurements of voltage at the sample and at the transformer output. Voltage and waveform data appearing on the oscilloscope screen were transferred to a PC through an HPIB (IEEE 488) parallel interface bus by means of HP Benchlink software. The data then could be stored for further analysis as needed (MS-Excel was used).

Test procedure

Surimi paste. Each sample was heated at five frequency levels (55 Hz, 500 Hz, 5 kHz, 50 kHz, and 200 kHz), each at 20, 40, 60, and 70°C. We started with 55 Hz to avoid interference from 60Hz electrical wiring and lights and assumed sample properties would be identical at 55 and 60 Hz. Because the Benchlink software could not continuously download and save measurements, each test had to be interrupted following measurements at each temperature level, to allow transfer of data to the PC. The voltage level from the transformer output was ~60V rms (giving a 120V differential). Current density through the sample increased with sample temperature; maximum value was ~3,500 A/m^2. The total heating time for each individual sample was about 1.5 min. To minimize electrode corrosion at low frequency (55 Hz), we used a lower voltage level (~40V rms or a drop across the sample of 80V) and current density (~2,300 A/m^2). Thus the test time at lower frequency was longer —about 4 min. Four replicates were measured for each composition at each frequency. For each repli-

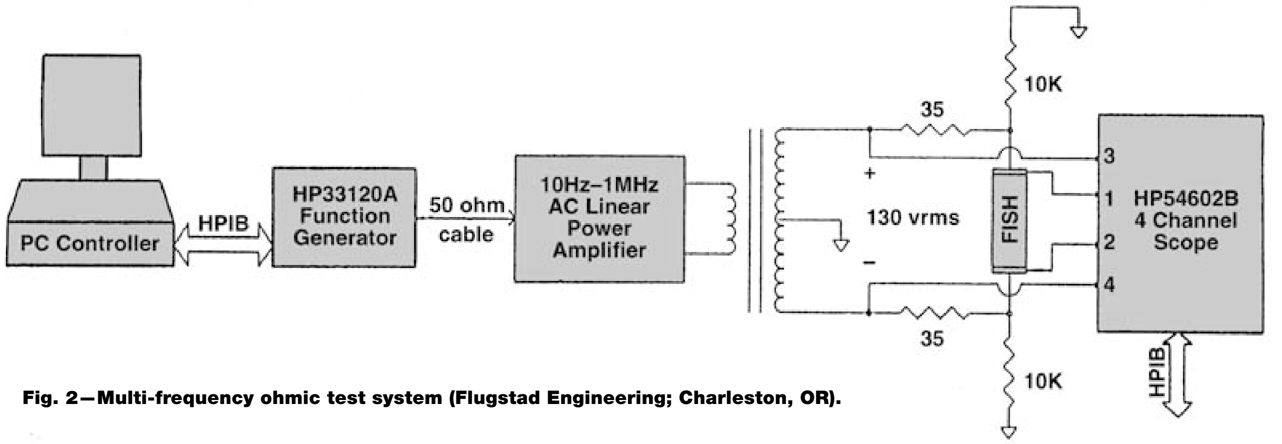

Fig. 2—Multi-frequency ohmic test system (Flugstad Engineering; Charleston, OR).

cate, the sample length was recorded for later electrical property calculation. The sample lengths among replicates were kept as nearly equal as possible during sample loading by adjusting to a mark on the electrode holders. By separate experiments, uncooked sample length could be controlled to within a standard deviation of ±0.5%. The average sample length increased 2.6% upon heating to 60°C; s.d. increased to ±1.2%.

Stabilized mince. Measurements at five frequencies (55 Hz, 500 Hz, 5 kHz, 50 kHz, and 200 kHz) were recorded for individual samples held at each of four temperatures (20, 40, 60, and 70°C). Each individual sample was heated to, and held at one test temperature while recording data for all five frequency levels. Then a different individual sample was tested at the next temperature at the same five frequency levels. The voltage level from the transformer output was ~60V rms (ΔV=120V). Current density through the sample increased with temperature, reaching a maximum of ~2,300 A/m^2. A lower voltage level (approximately 40 ΔV rms, or ΔV=80) was used for the low frequency value, 55 Hz, to minimize electrode corrosion. The time required to test each sample over all frequency levels was about 10 min. This procedure was repeated for four replicates. While testing at a single temperature, the applied frequencies began with the highest (200 kHz) and ended with the lowest (55 Hz). This minimized the time electrodes were exposed to the low frequency condition at which corrosion was notable. No corrosion was observed for frequencies >5 kHz.

Determination of R_{dc} and R_e

From the parallel resistor model (Fig. 1), we anticipate that sample impedance Z_{load} and resistance R_{dc} would be equal at low frequencies. At high frequencies, Z_{load} would become less than R_{dc} if R_e and C_{fish} had finite values. Early tests showed a sudden rise in impedance at low frequencies, due apparently to electrode corrosion. To negate this corrosion effect, a straight line regression based on the experimental values of Z_{load} at frequency values >5 kHz was used to simulate the sample impedance behavior. The extension of this line to the lowest frequency (55 Hz, assumed to be the DC condition), would be taken from the linear regression curve. DC electrical conductivity σ_{dc} was calculated from the electrical resistance R_{dc}, the Z_{load} at the DC condition. Because no capacitive effect was found for any case, R_e could be calculated from knowledge of Z_{load} and R_{dc} (Fig.1). Then the value of σ_{dc} and ϵ''could be calculated from equations given earlier.

RESULTS & DISCUSSION

IN EARLY EXPERIMENTS WITH SURIMI paste at 55 Hz, we had found that values of σ_{dc} were about 15% higher and considerably less variable when measured under applied pressure, compared to those measured without applied pressure. This was possibly due to air voids in the sample. Thus, an experimental method using applied pressure was used in the current study.

In the low frequency range (<500 Hz), dielectric properties were independent of applied frequencies. In low frequency ohmic heating of foods, only DC electrical conductivity σ_{dc} determines the food impedance and therefore the rate of heat generated. Impedance Z_{load} decreased with frequency. However, obvious for both surimi paste and stabilized mince, no phase shift was observed between the waveform of voltages across the sample and those from the transformer outputs. This indicated no electrical capacitance (or effect of absolute dielectric constant ϵ'). Thus the sample impedance Z_{load} was based only on the resistances R_{dc} and R_e (the effect of absolute dielectric loss ϵ'').

Surimi paste

The sample impedance Z_{load} decreased with applied frequency (Fig. 3a) for Pacific whiting surimi paste. The resulting values of σ_{dc} changed with respect to temperature at various levels of salt concentration (Fig. 3b).

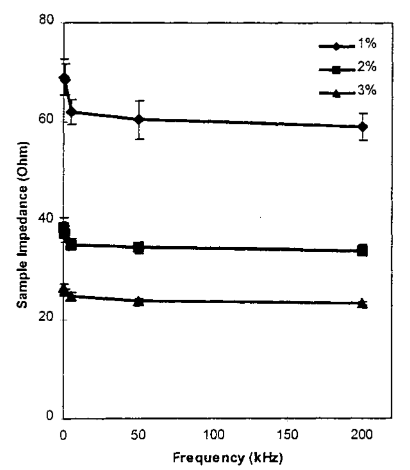

Fig. 3a—Representative data giving sample impedance of surimi paste as a function of frequency and salt. 79% moisture content; 40°C; salt concentration: 1% (♦); 2% (■); 3% (▲) .

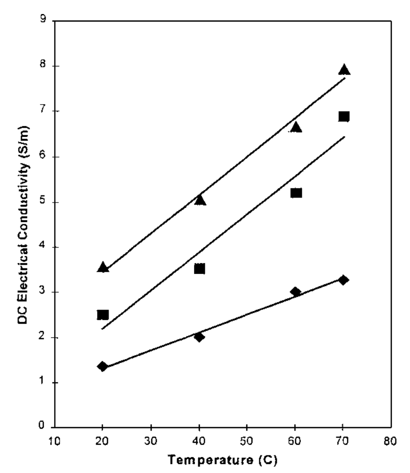

Fig. 3b—Representative data and straight line regression, showing DC electrical conductivity of surimi paste as a function of temperature and salt. 79% moisture content; salt concentration: 1% (♦); 2% (■); 3% (▲).

The curves shown represent the general finding, that DC electrical conductivity increased with temperature and salt concentration.

An empirical model was derived ($r^2 = 0.965$) for DC electrical conductivity of Pacific whiting surimi paste as a function of temperature and salt concentration:

$$\sigma_{dc} = -0.337 + 0.026T + 57.89S + 2.433TS$$

where σ_{dc} = DC electrical conductivity (S/m); T = temperature (°C); S = salt concentration (fraction w/w).

Positive coefficients of temperature and salt concentration indicated that DC electrical conductivity increased with both. Furthermore, the positive coefficient of TS indicated that electrical conductivity of a sample with any salt concentration would increase with temperature. The model predicted Pacific whiting surimi paste DC electrical conductivity with an error ranging from –16.5% to 8.3%. There was a difference of –6.4%~14.7% between the prediction based on our model and that previously described by Yongsawatdigul et al. (1995a). The absolute dielectric loss, ϵ'' of Pacific whiting surimi paste changed with temperature at various levels of salt concentration (Fig. 3c). Salt ions act as conductive charge carriers and increase the absolute dielectric loss ϵ'' (Datta et al., 1995).

An empirical model was derived ($r^2 = 0.808$) for absolute dielectric loss ϵ'' of Pacific whiting surimi paste as a function of temperature and salt concentration:

$$\epsilon'' = 0.302 - 1.8 \times 10^{-2}T - 4.94S + 0.45TS + 1.92 \times 10^{-4}T2$$

where ϵ'' = absolute dielectric loss (mF/m); T = temperature (°C); S = salt concentration (fraction w/w).

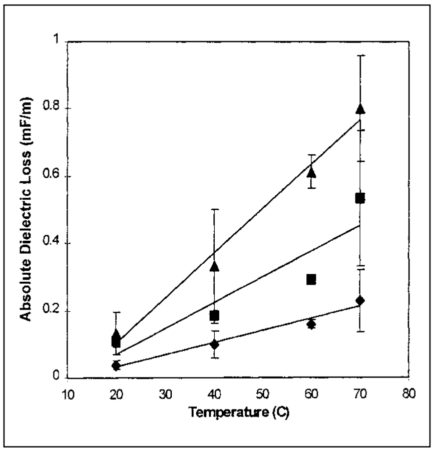

Fig. 3c—Representative data and straight line regression showing dielectric loss factor of surimi paste as a function of temperature and salt. 79% moisture content; salt concentration: 1%(◆); 2%(■); 3%(▲).

Stabilized mince

Because only one sample composition of stabilized mince was considered, the DC electrical conductivity σ_{dc} and absolute dielectric loss ϵ'' of stabilized mince were measured as functions of temperature only. The sample impedance Z_{load} decreased with applied frequency (Fig. 4); analysis followed the procedures used for surimi paste samples. DC electrical conductivity increased with temperature (Fig. 5) and can be represented by the following model with $r^2 = 0.97$ and error range of -3.33% to 4%:

$$\sigma_{dc} = 0.262 + 0.0193T$$

where σ_{dc} = DC electrical conductivity (S/m); T = temperature (°C).

The absolute dielectric loss ϵ'' also increased with temperature (Fig. 6) with a linear model having an r^2 of 0.973 and error range of -16 to 1%:

$$\epsilon'' = -1.43 \infty 10^{-2} + 2.3 \infty 10^{-3}T$$

where ϵ'' = absolute dielectric loss (mF/m); T = temperature (°C).

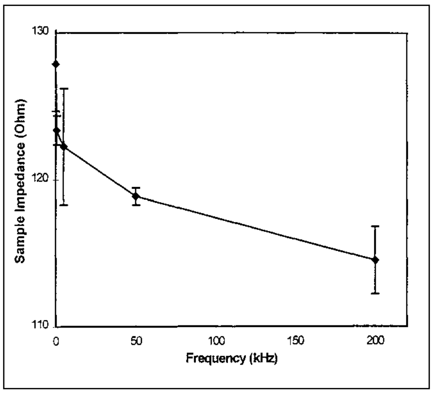

Fig. 4—Representative data giving sample impedance of stabilized mince as a function of frequency. 77% moisture content; 0.74% salt concentration; 40°C.

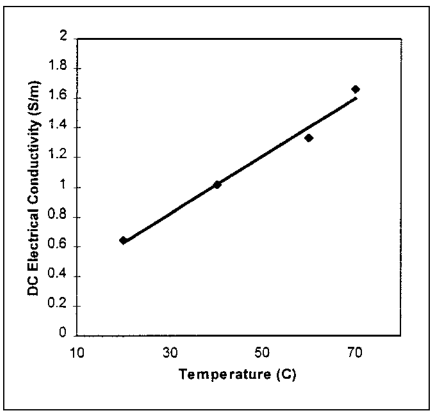

Fig. 5—Representative data and straight line regression giving DC conductivity of stabilized mince as a function of temperature. 77% moisture content; 0.74% salt concentration.

Note that the heating procedure for stabilized mince involved a frequency sweep for samples held at a constant temperature. For surimi paste, each sample underwent a temperature sweep while held at a single frequency. Unlike surimi paste, unwashed and stabilized mince has a less consistent composition and contains a protease concentration on the order of ten times that of surimi (An et al., 1994a, b). The additional enzymes Cathepsin B and Cathepsin H are very active at 20°C~40°C and tended to distort the measured data, occasionally indicating physically impossible results. Ideally, one sample would be used for all temperature and frequency levels to minimize sample variance. However, such a test would take a long time (~45~60 min), allowing the protease to degrade the protein network, possibly changing sample properties. For stabilized mince, test time was minimized by quickly heating to the target temperature and sweeping frequencies.

Corrosion of electrode surface

An electrolytic reaction could take place at the electrode surface while conducting 60 Hz ohmic heating tests with current density exceeding 3,500 A/m^2 and sample salinity up to 4% (Shiba, 1992; Yongsawatdigul et al.,1995a; Reznik, 1996). Such a reaction would cause a burned product and corrosion of the electrode. Our study experienced this problem with all samples tested at 55 and 500 Hz (with current density up to 3,000 A/m^2). The corrosion appeared as a light brown porous film in an irregular pattern at low salt concentrations (1%), which became more uniform at higher salt concentrations. As frequency exceeded 500 Hz, we observed that corrosion diminished, as reported by others (Uemura et al., 1994; Reznik, 1996). When conducting experiments on Pacific whiting stabilized mince samples at 5 kHz and higher, the

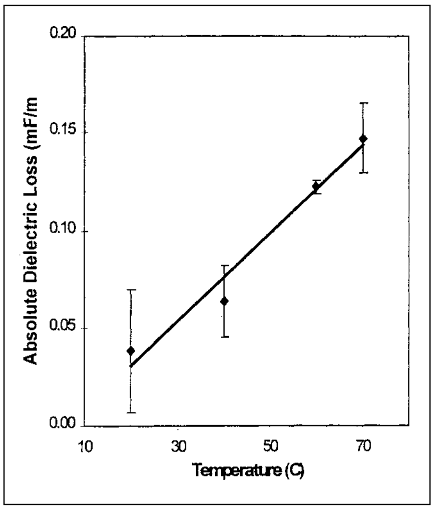

Fig. 6—Representative data and straight line regression giving dielectric loss of stabilized mince as a function of temperature. 77% moisture; 0.74% salt concentration.

corrosion on the electrode surface and burning of the sample were no longer visible.

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