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Dielectric properties of salmon (*Oncorhynchus keta*) and sturgeon (*Acipenser transmontanus*) caviar at radio frequency (RF) and microwave (MW) pasteurization frequencies

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Abstract

Radio frequency (RF) and microwave (MW) heating provide an important advantage of more rapid heat penetration in pasteurization processes for heat labile high value foods, which to date, have only been pasteurized by conductive heating. The objectives of this work were to determine the dielectric constant, loss factor and power penetration depth for salmon (0.8% and 2.3% total salt) and sturgeon (0.20 and 3.3% salt) caviars at RF frequency of 27 MHz and MW frequency of 915 MHz (20–80 °C). The dielectric constant (ε') and dielectric loss factor (ε'') for salmon and sturgeon caviar increased markedly with increasing temperature at 27 MHz but not at 915 MHz. Power penetration depth was higher at 27 MHz compared to 915 MHz, and in unsalted compared to salted roe. Power penetration depth tended to decrease as temperature increased.

Keywords: Dielectric properties; Caviar; Salt; Temperature; Dielectric constant; Dielectric loss; Power penetration depth

1. Introduction

Caviars are ready to eat aquatic food products made by brining and curing fish roe (Bledsoe, Bledsoe, & Rasco, 2003). Over 90% of the salmon caviar or ikura in USA is produced in the Pacific Northwest ($350 million per year). There are more than 20 species of sturgeon harvested for caviar, with major production and resource remediation efforts focused on *Huso* sp. and *Acipenser* sp. in the former Soviet Union, Eastern Europe, China and the United States. There is an increasing production of 'black caviars' from cultured sturgeon and other species such as paddlefish (*Polyodon spathula*), in part to relieve fishing pressure on threatened and endangered *Huso* sp. (Department of Interior, 2002). US domestic production of sturgeon caviar in 2000 was 60,000 pounds or roughly one-third of US consumption (Bledsoe et al., 2003; Passy, 2001). Average retail prices are $1250/kg for US domestic product.

Refrigerated storage is currently the only available means to preserve and extend the shelf life of these valuable products. Yet, refrigeration alone certainly cannot assure a pathogen-free product with a long shelf life. International markets are demanding pasteurized products as a means of reducing the risk of food borne illness. Caviar is heat labile and difficult to pasteurize because of its sensitivity to heat treatment. Irreversible protein denaturation occurs at temperatures slightly above 80 °C (Sternin, 1992). Radio frequency (RF) and microwave (MW) heating provides a possible

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alternative to the conventional thermal processing for caviar products. Dielectric heating processes have the advantage of shortening process time and often yield higher quality products than those produced using conventional thermal processing methods (Guan, Plotka, Clark, & Tang, 2002). A radio frequency-based sterilization heating process has been suggested for low acid foods such as macaroni and cheese. In addition to the shorter heating time, less changes in the quality attributes of the product were noticed in the RF treated product compared to the retort-sterilized product (Wang, Wig, Tang, & Hallberg, 2003).

Batch pasteurization of sturgeon and salmon caviars in airtight or vacuum packaged containers (50–70 °C) have been attempted, but with limited success (Bledsoe et al., 2003; Sternin, 1992). Pasteurized salmon caviar becomes soft and pale, and immature eggs loose their shape at 71 °C, eggs appear dull, and at 72 °C, the egg yolk is completely coagulated and salmon caviar is transformed into a chewy mass (Sternin & Dore, 1993).

Dielectric properties are a measure of how food interacts with electromagnetic energy during dielectric heating. Knowing the dielectric properties of a food is required to design a dielectric heating process since these parameters affect coupling and distribution of electromagnetic radiation during the heating process (Mudgett, 1986). Many variables in food systems affect RF or MW heating performance. The dielectric constant (ε') is the characteristic of the material that describes its ability to absorb, transmit and reflect energy from the electric portion of RF or MW waves. The dielectric loss factor (ε'') describes how well a material absorbs energy from electric fields passing through it and converts that energy to heat. A third important concept in the dielectric heating is the power penetration depth (Buffler, 1993), defined as the distance an incident electromagnetic wave can penetrate beneath the surface of a material as the power decreases to $1/e$ of its power at the surface. The interaction between the ε' and ε'' determine the distance over which the power decreases by a certain factor and therefore determine the penetration depth (Tang, Feng, & Lau, 2002).

The dielectric properties of foods are often temperature dependent (Tang et al., 2002), therefore, understanding the effect a wide range of temperatures has on the dielectric properties of biological materials is vitally important to properly predicting heating behavior. The effect of temperature on dielectric properties of different foods has been reviewed (Bengtsson & Risman, 1971; Tang et al., 2002). Other factors such as salt content plays an important role in controlling the dielectric properties of food (Herve, Tang, Luedecke, & Feng, 1998). Salmon caviars range from 20–38% protein and 10–20% crude lipid (Bledsoe et al., 2003; Huang et al., 2001) with a water activity of 0.96–0.98 and moisture content of 50–56%, pH of 5.5–6.7 and a salt content ranging from 2–5% (Bledsoe et al., 2003). Sturgeon caviar, on the other hand has 17–32% protein, 11–18% crude lipid, with moisture content range between 57–77% and water activity of 0.95. Salted sturgeon caviars typically have a salt content of 3–3.5% (Bledsoe et al., 2003).

The objectives of this study were to determine the dielectric properties of untreated sturgeon and salmon caviar; study the effect of product temperature (20–80 °C) on dielectric properties in connection with a potential use in the development of pasteurization protocols; and to investigate the impact of commercially used salt concentrations on the dielectric properties.

2. Materials and methods

2.1. Equipment and materials

An Agilent (formerly Hewlett Packard) 4291 B Impedence material analyzer (Agilent Technologies, Palo Alto, CA), with an open-ended coaxial probe and a custom built test cell, a VWR Model 1157 programmable circulator (VWR Science Products, West Chester, PA) (Wang et al., 2003) were used to measure the dielectric constant (ε') and dielectric loss factor (ε''). The 4291 B impedance analyzer can make measurements over the frequency range of 1–1800 MHz. The probe was rated for use in the temperature range of −40 to +200 °C. The impedance analyzer was connected through an IEEE-488 (GPIB) bus to a desktop personal computer, which is used with custom-designed software DMS 85070 (Innovative Measurements Solutions), controlled the impedance analyzer and logged measured data. Approximately 15 g samples of green (unsalted) and salted sturgeon and salmon caviar were used. Salmon roe (*Onorhynchus keta*) was obtained from the Southern Southeast Regional Aquaculture Association (Ketchikan, AK) and from Mayco Fish Co., Tacoma, WA. Sturgeon roe (*Acipenser transmontanus*) was kindly donated by Stolt Sea Farms, Elvira, CA. The eggs were crushed into a homogeneous dispersion with air pockets removed prior to measurement.

2.2. Calibration and frequency dependence

Before the measurements the impedance analyzer was warmed up for at least 30 min, following the recommendations of the manufacturer. A 4219 B calibration kit that included four calibration standards: an open, a short, a 50 Ω load, and a low-loss capacitor, was used to calibrate the impedance analyzer. Thereafter, the testing probe was calibrated using an 85070B dielectric probe kit, which included a short circuit (a gold-plated precision shorting block), an open circuit (air), and a known load (pure water at 25 °C). After the calibration, a sample was placed into a custom-built temperature-controlled test cell

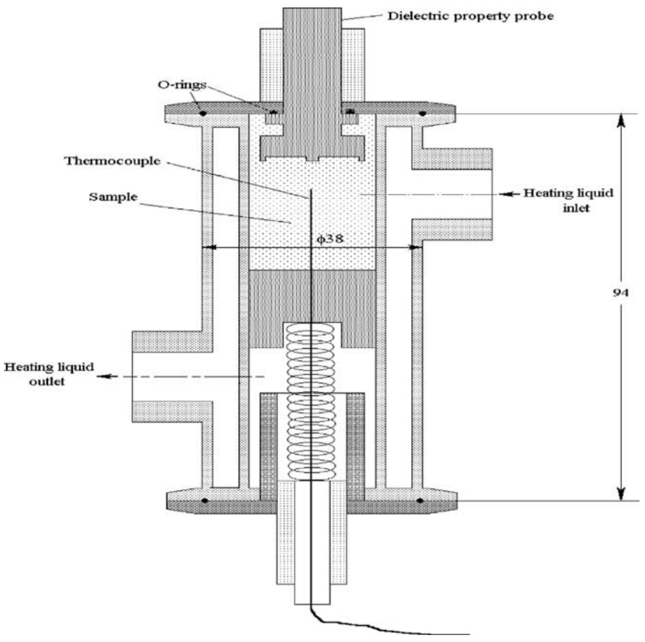

Fig. 1. Diagram of pressure-proof dielectric test cell (stainless steel), dimensions in mm (Wang et al., 2003).

(Fig. 1). Measurements were conducted every 10 °C from 20 to 80 °C and from 1.0 to 1800.0 MHz. A minimum of 10 min was used to permit temperature re-equilibration after each temperature adjustment. Prior to measurement, samples were conditioned in a water bath at the desired temperatures (±0.1 °C). Sample temperature was verified using a digital thermometer (Barnatt 115, Model 600–1020, Barrington, IL).

2.3. Measurements of penetration depth

Sodium chloride concentration was determined by AOAC method 973.09 (AOAC, 1995). Green or untreated salmon and sturgeon caviar were found to contain 0.8% and 0.2% (w/w) sodium chloride, respectively. Salted sturgeon and salmon caviar contained approximately 2.3% and 3.3%, respectively. Dielectric properties were measured in triplicate samples at each temperature. Power penetration depth was calculated according to the following equation:

$$dp = \frac{C}{2\sqrt{2}\pi f\left\{\varepsilon'\left[\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right]\right\}^{\frac{1}{2}}} \qquad (1)$$

dp: power penetration depth, m; *C*: the speed of light in vacuum, 2.998×10^8 m/s; *f*: temporal frequency, Hz; ε': dielectric constant; ε'': dielectric loss factor.

3. Results and discussion

Water and salt content strongly influence the dielectric properties of a food. Water is a dipolar compound that couples more dielectric energy than most other food components, therefore, any endogenous or added food constituents that bind water could affect the dielectric properties of a food. The dielectric constant and loss factor of salted and unsalted sturgeon caviar were determined at 27 MHz and 915 MHz at a temperature range of 20–80 °C. Figs. 2–5 show that the ε' and ε'' for salted and unsalted caviar from either sturgeon or salmon are frequency and temperature dependent. The values of ε' and ε'' increased as the temperature increased, with this trend being more pronounced at 27 MHz for samples at the higher salt content.

At 27 MHz, the pronounced increase in ε' with increasing temperature is probably in response to an increased degree of dielectric dispersion due to ionic conductivity as temperature rises (Nelson & Bartley, 2001). This phenomenon may have also contributed to the increase in the ε'' as temperature rises. At 915 MHz, the ε' increased mildly as the temperature increased for both salted and unsalted sturgeon caviar and remained relatively constant for salmon caviar (Figs. 2 and 3) due to relatively low moisture content (50–56% for salmon

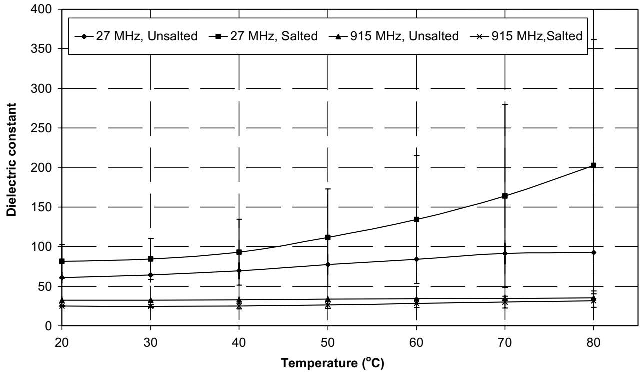

Fig. 2. Dielectric constant for salted (3.3%) and unsalted (0.2%) sturgeon caviar at 27 MHz and 915 MHz.

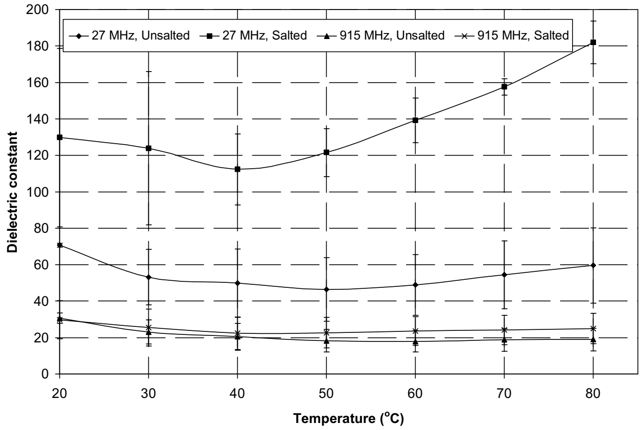

Fig. 3. Dielectric constant for salted (2.3%) and unsalted (0.8%) salmon caviar at 27 MHz and 915 MHz.

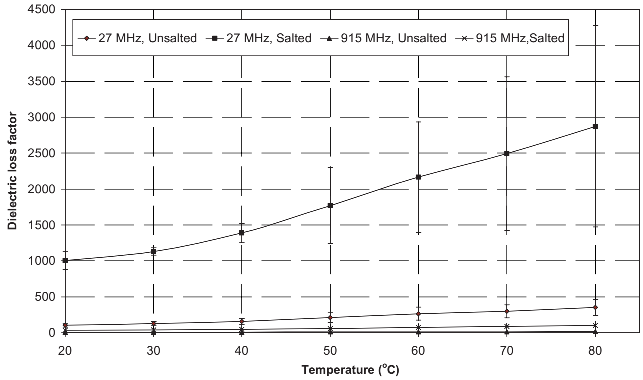

Fig. 4. Dielectric loss factor for salted (3.3%) and unsalted (0.2%) sturgeon caviar at 27 MHz and 915 MHz.

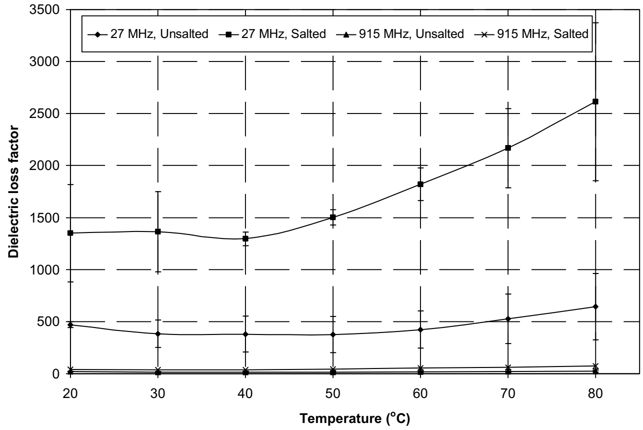

Fig. 5. Dielectric loss factor for salted (2.3%) and unsalted (0.8%) salmon caviar at 27 MHz and 915 MHz.

caviar, and 57–77% for sturgeon caviar) (Bledsoe et al., 2003).

There are some discrepancies in the literature pertaining to the impact of temperature as a parameter for the dielectric properties of a food material. Some researchers have found that dielectric constant decreases with increasing temperature (Brican & Barringer, 2002; Ohlsson & Bengtsson, 1975; Ohlsson, Bengtsson, & Risman, 1974). Other systems where ε′ has been found to increase with temperature include: milk and whey powders (Rzepecka & Pereria, 1974) potato starch and carboxymethycellulose powder (Nelson, Prakash, & Lawerence, 1991). Whether the ε′ of a food increases or decreases is not only affected by temperature but also affected by frequency and moisture content. Normally at low frequencies, dielectric constant increases with increasing temperature. At high frequencies, dielectric constant decreases with increasing temperature if the moisture content is high (>70%); increases with increasing temperature if the moisture content is low (<70%). The results in this study agreed with the trend reported by Wang et al. (2003). They indicated that dielectric constants of cooked macaroni noodles increased with temperature in the radio frequency range (27 and 40 MHz) and also increased with temperature in the microwave range (915 and 1800 MHz) due to the relatively lower water content (56%) of the cooked macaroni noodles. Feng, Tang, and Cavalieri (2002) also reported that dielectric constants of Red Delicious apples (*Malus domestica Borkh*) increased with temperature in the microwave range (915 and 1800 MHz) when water content of apples was less than 70%, and decreased when water content was more than 70%. In ham, the ε′ of samples with low moisture and high ash contents (for example, 45.0% moisture, 4.65% ash) increased with temperature at 2450 MHz (Sipahioglu, Barringer, Tauba, & Prakash, 2003).

Dielectric loss factor is composed of two components: dipole loss and ionic loss. Dipole loss results from the rotation of water dipoles, while ionic loss results from migration of ions. Generally dipole loss decreases with temperature, while the ionic loss component increases with temperature at the microwave frequencies (915 and 2450 MHz) (Sipahioglu et al., 2003; Tang et al., 2002; Wang et al., 2003). Moreover, the ionic conductivity predominantly affects the ε″ at frequencies below 200 MHz (Guan, Cheng, Wang, & Tang, 2004; Wang et al., 2003). Electronic conduction and various polarization mechanisms, including dipole, electronic, atomic, and Maxwell–Wanger contribute to the ε″ (Tang et al., 2002). In the presence of water, salt ionizes and migrates with the changing electromagnetic field direction (Mudgett, Goldblith, Wang, & Westphal, 1980). Moisture greatly affects ε″, however, salt was the major contributing factor in the noticeable upsurge in the ε″ as the temperature increased from 20 to 80 °C for salted sturgeon and salmon caviar (Figs. 4 and 5). For unsalted sturgeon and salmon caviar, the main contributer to ε″ was most likely dipole loss (Figs. 4 and 5).

The results obtained in this study for ε″ support those of Wang et al. (2003) showing that the ε″ of protein gels and liquid whey protein mixture increased sharply at 27 and 40 MHz with increasing temperature, but only slightly increased at 915 and 1800 MHz. The large standard deviation of the ε′ and ε″ observed at 27 MHz is possibly because the salmon and sturgeon caviar samples tended to change phase from a semi-solid dispersion to solid mass resulting from protein denaturation as the temperature approached 80 °C. Additionally, the presence of collagenous material in the egg shell might have lead to the large standard deviation. The high values for ε″ at 27 MHz at high temperatures may also have resulted from reduced material viscosity leading to increased mobility of ions and a higher conductivity (Tang et al., 2002). However at higher frequency (915 MHz), little change in the ε″ was observed as the temperature increased suggesting that ionic conduction that predominates at lower frequencies was offset by the dipole rotation of water molecules, an effect that becomes more prominent at higher frequencies. In addition, at elevated temperatures, hydrogen bonds formation rate may be lower depressing ε'' value as temperature increases (Ohlsson, 1989).

In general, the greater the moisture and salt content of a food, the shallower the penetration depth, and consequently the less even the heating rate throughout the food (Nelson et al., 1991). The power penetration depths in sturgeon and salmon caviar were greater at 27 MHz. It was 4–5 times greater than at 915 MHz regardless of salt content suggesting that it is possible to achieve more uniform heating characteristics when caviar is heated at 27 MHz (Tables 1 and 2, and Figs. 6–9).

The power penetration depth decreased with increasing temperature, with a depression in penetration depth

Table 1
Dielectric properties and power penetration depths (mean ± SD)[a] for salted (3.3%) and unsalted (0.2%) sturgeon (*Acispencer transmontanus*) caviar

T (°C)		27 MHz			915 MHz		
		ε'[b] ± SD	ε''[c] ± SD	pd[d] ± SD	ε' ± SD	ε'' ± SD	pd ± SD
20	Unsalted	61.0 ± 7.0	105.5 ± 24.8	16.0 ± 1.7	32.6 ± 4.1	8.9 ± 1.5	3.4 ± 0.4
	Salted	81.5 ± 21.2	1004.0 ± 127.8	4.2 ± 0.4	25.0 ± 0.4	35.8 ± 0.4	0.9 ± 0.0
30	Unsalted	64.2 ± 9.2	128.1 ± 31.2	14.2 ± 1.5	32.6 ± 3.6	9.0 ± 1.3	3.4 ± 1.3
	Salted	84.6 ± 25.6	1130.5 ± 51.1	3.8 ± 0.1	24.9 ± 1.9	41.7 ± 3.6	0.8 ± 0.0
40	Unsalted	69.8 ± 10.3	158.4 ± 38.5	12.5 ± 1.3	33.0 ± 3.6	9.9 ± 1.3	3.1 ± 0.2
	Salted	93.0 ± 41.6	1387.8 ± 135.7	3.5 ± 0.3	25.0 ± 3.7	48.0 ± 12.1	0.7 ± 0.1
50	Unsalted	77.4 ± 12.8	210.8 ± 68.2	11.0 ± 1.6	33.7 ± 4.4	11.3 ± 2.0	2.7 ± 0.3
	Salted	111.5 ± 61.5	1769.5 ± 528.7	3.1 ± 0.4	26.4 ± 4.7	59.5 ± 22.9	0.6 ± 0.2
60	Unsalted	84.0 ± 14.5	266.4 ± 91.9	9.8 ± 1.6	34.0 ± 4.7	13.9 ± 3.3	2.4 ± 0.3
	Salted	134.3 ± 80.7	2165.0 ± 770.7	2.8 ± 0.5	28.4 ± 5.4	73.0 ± 32.4	0.6 ± 0.2
70	Unsalted	91.6 ± 13.2	297.8 ± 91.8	8.6 ± 1.4	34.7 ± 4.5	15.1 ± 3.1	2.1 ± 0.3
	Salted	164.0 ± 115.7	2491.9 ± 1069.0	2.7 ± 0.5	30.2 ± 7.4	86.1 ± 45.1	0.5 ± 0.2
80	Unsalted	92.5 ± 13.4	352.2 ± 109.4	7.8 ± 1.4	35.3 ± 3.3	17.0 ± 3.3	1.9 ± 0.2
	Salted	202.8 ± 158.8	2873.3 ± 1401.6	2.5 ± 0.6	31.9 ± 8.6	99.9 ± 58.1	0.5 ± 0.2

[a] Results are the mean values of at least three independent trials ($N = 3$).
[b] ε' is dielectric constant.
[c] ε'' is dielectric loss factor.
[d] pd is penetration depth.

Table 2
Dielectric properties and power penetration depths (mean ± SD)[a] for salted (2.3%)and unsalted (0.8%) salmon (*Oncorhynchus keta*) caviar

T (°C)		27 MHz			915 MHz		
		ε'[b] ± SD	ε''[c] ± SD	pd[d] ± SD	ε' ± SD	ε'' ± SD	pd ± SD
20	Unsalted	70.7 ± 1.1	470.8 ± 27.6	6.3 ± 0.2	30.7 ± 2.9	18.7 ± 1.5	1.7 ± 0.1
	Salted	129.8 ± 49.0	1349.4 ± 466.1	3.7 ± 0.6	29.8 ± 10.4	40.5 ± 17.2	0.9 ± 0.2
30	Unsalted	53.2 ± 15.2	383.6 ± 131.5	7.0 ± 1.3	23.1 ± 6.7	15.0 ± 5.0	1.8 ± 0.3
	Salted	123.9 ± 42.1	1363.4 ± 385.0	3.6 ± 0.5	25.5 ± 10.2	38.3 ± 17.8	1.0 ± 0.4
40	Unsalted	49.9 ± 18.8	379.5 ± 173.2	7.2 ± 1.8	20.5 ± 7.4	14.5 ± 6.4	1.9 ± 0.5
	Salted	112.3 ± 19.5	1296.7 ± 66.0	3.7 ± 0.1	22.4 ± 8.9	36.6 ± 14.5	0.9 ± 0.2
50	Unsalted	46.4 ± 17.5	375.9 ± 173.2	7.2 ± 1.8	18.3 ± 6.3	14.1 ± 6.3	1.8 ± 0.5
	Salted	121.5 ± 13.1	1501.1 ± 73.8	3.4 ± 0.1	22.7 ± 8.4	43.3 ± 13.6	0.7 ± 0.1
60	Unsalted	48.9 ± 16.7	423.6 ± 180.5	6.7 ± 1.5	17.9 ± 5.9	15.6 ± 6.5	1.6 ± 0.4
	Salted	139.2 ± 12.2	1820.6 ± 156.3	3.0 ± 0.1	23.6 ± 7.8	52.5 ± 14.9	0.7 ± 0.1
70	Unsalted	54.5 ± 18.6	525.5 ± 237.2	6.0 ± 1.4	18.7 ± 6.2	18.9 ± 8.3	1.4 ± 0.4
	Salted	157.6 ± 4.5	2168.4 ± 381.1	2.8 ± 0.3	24.2 ± 8.1	61.5 ± 19.7	0.6 ± 0.1
80	Unsalted	59.6 ± 20.6	642.7 ± 318.1	5.5 ± 1.5	18.9 ± 6.2	22.2 ± 10.2	1.3 ± 0.4
	Salted	182.0 ± 11.7	2614.5 ± 758.9	2.6 ± 0.4	25.0 ± 8.4	73.6 ± 26.3	0.5 ± 0.1

[a] Results are the mean values of at least three independent trials ($N = 3$).
[b] ε' is dielectric constant.
[c] ε'' is dielectric loss factor.
[d] pd is penetration depth.

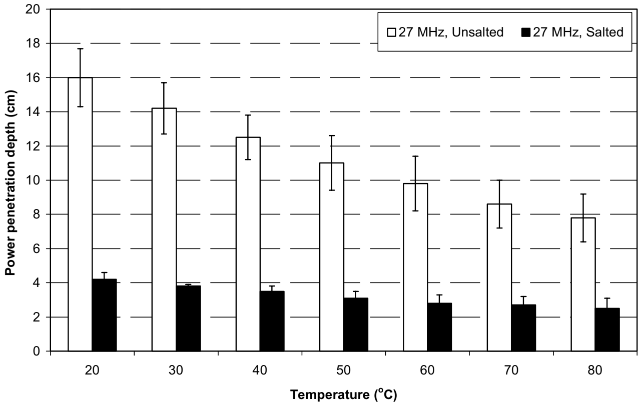

Fig. 6. Power penetration depths of electromagnetic waves in salted (3.3%) and unsalted (0.2%) sturgeon caviar at 27 MHz.

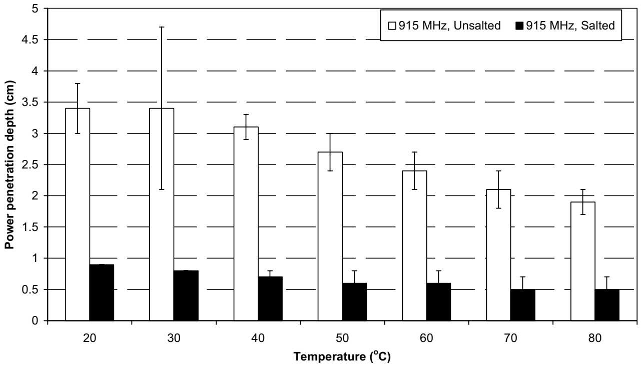

Fig. 7. Power penetration depths of electromagnetic waves in salted (3.3%) and unsalted (0.2%) sturgeon caviar at 915 MHz.

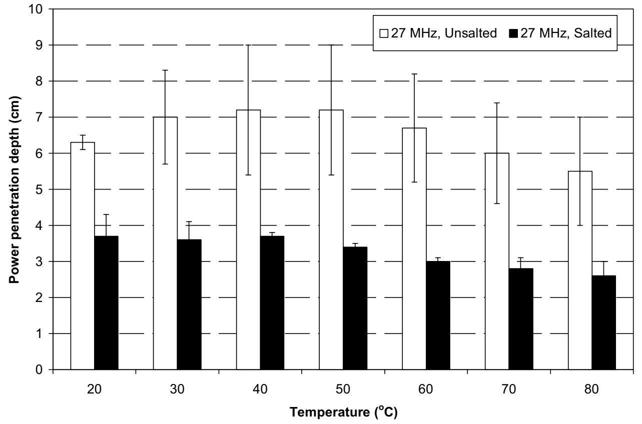

Fig. 8. Power penetration depths of electromagnetic waves in salted (2.3%) and unsalted (0.8%) salmon caviar at 27 MHz.

further accentuated in the products containing salt, probably due to the high ε'' induced by a higher salt content especially at high temperatures. The power penetration depths in unsalted sturgeon caviar (20 °C) were 16 cm at 27 MHz compared to only 3.4 cm at 915 MHz. In comparison, the pentration depth in salted sturgeon

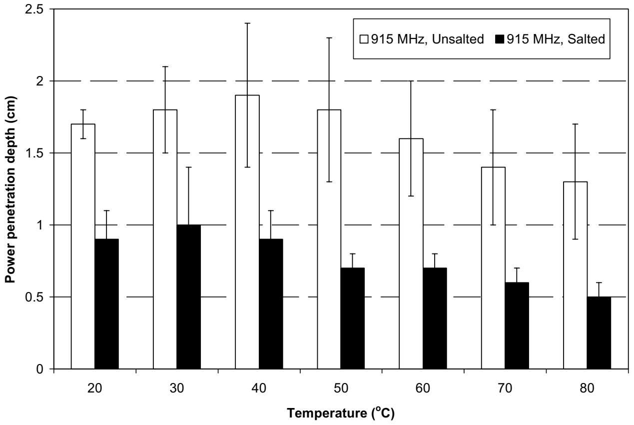

Fig. 9. Power penetration depths of electromagnetic waves in salted (2.3%) and unsalted (0.8%) salmon caviar at 915 MHz.

caviar dropped almost 3/4 compared to the unsalted product at the same temperature. Temperature had a lower impact on the penetration depth compared to salt and increasing the temperature from 20 to 80 °C reduced the penetration depth roughly by 1/2 (Table 1). The relatively greater change in penetration depth observed for sturgeon compared to salmon caviar was probably due to the higher salt concentration.

4. Conclusions

The dielectric properties (ε' and ε'') of valuable sturgeon and salmon caviar products were measured and were higher at 27 MHz compared to 915 MHz. A large increase in ε' and ε'' was observed with increasing temperature and salt content. Changing frequency from the RF (27 MHz) to microwave (915 MHz) had a greater affect on the penetration depth (4–5 times) than changing the temperature from 20 to 80 °C (50%). The results obtained in this study along with results obtained from thermobacteriological studies provide the basis for designing a dielectric pasteurization protocol for salmon and sturgeon caviar to produce a safe, high quality product.

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