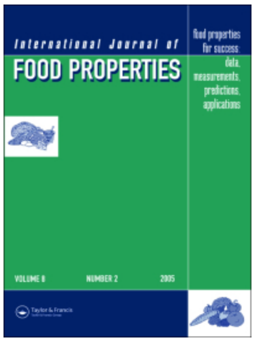

International Journal of Food Properties

ISSN: 1094-2912 (Print) 1532-2386 (Online) Journal homepage: www.tandfonline.com/journals/ljfp20

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To cite this article: Ruth Hernández-Nava, Juan Mateo Meza-Arenas, Diego Sarmiento-Narvaez, Tejinder Kaur, Alonso Corona-Chavez, Roberto Rojas-Laguna & María Elena Sosa-Morales (2025) Dielectric properties of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.) at different temperatures for radio and microwave frequencies, International Journal of Food Properties, 28:1, 2489489, DOI: 10.1080/10942912.2025.2489489

To link to this article: https://doi.org/10.1080/10942912.2025.2489489

Published online: 09 Apr 2025.

INTERNATIONAL JOURNAL OF FOOD PROPERTIES
2025, VOL. 28, NO. 1, 2489489
https://doi.org/10.1080/10942912.2025.2489489

Dielectric properties of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.) at different temperatures for radio and microwave frequencies

Ruth Hernández-Nava[a], Juan Mateo Meza-Arenas[b], Diego Sarmiento-Narvaez[b], Tejinder Kaur[c], Alonso Corona-Chavez[b], Roberto Rojas-Laguna[d], and María Elena Sosa-Morales[e]

[a]Departamento de Ingeniería Química, Alimentos y Ambiental, Universidad de las Américas Puebla, San Andrés Cholula, Mexico; [b]Departamento de Electrónica, Instituto Nacional de Astrofísica, Óptica y Electrónica, Tonanzintla, Mexico; [c]Departamento de Sistemas Electrónicos y de Telecomunicaciones, Universidad Autónoma de la Ciudad de Mexico, CDMX, Mexico; [d]Departamento de Ingeniería Electrónica, División de Ingenierías, Campus Irapuato-Salamanca, Universidad de Guanajuato, Salamanca, Mexico; [e]Departamento de Alimentos, División de Ciencias de la Vida, Campus Irapuato-Salamanca, Universidad de Guanajuato, Irapuato, Mexico

ABSTRACT

The bulk permittivity (ε_{bulk}) and particle permittivity (ε_{part}) of in-shell and shelled sunflower seeds (*Helianthus annuus* L) and pine nuts (*Pinus pinea* L.) were determined in a temperature range of 20–60°C and a frequency range of 27–5000 MHz using the transmission line method. Additionally, the samples were analyzed for moisture content, water activity ($a_{w,}$), fat content, color, and densities (bulk, tapped, and particle). The dielectric constant (ε′) decreased with increasing temperature for sunflower seeds (from 3.04 to 2.63 for in-shell samples) and increased with temperature for pine nuts (from 4.41 to 5.48, also for in-shell seeds), due to the differences in the a_w values. For all samples, the ε′ and loss factor (ε′′) decreased with increasing frequency. ε″ and ε′′ values increased with higher bulk density; for instance, at frequency of 915 MHz, in-shell sunflower seeds ($\rho_{bulk} = 0.341$ g/cm^3) had ε″ = 1.49 and ε′′ = 0.02 at 20°C, while in-shell pine nuts ($\rho_{bulk} = 0.601$ g/cm^3) had ε″ = 2.53 and ε″ = 0.03 at 20°C. Higher penetration depth (d_p) values were found at lower frequencies, for example, shelled sunflower seeds at 60°C exhibited d_p = 7.52 at 27 MHz, but d_p = 1.20 m at 5000 MHz. Results are valuable for designing further radiofrequency and microwave treatments for these seeds and nuts.

ARTICLE HISTORY
Received 30 December 2024
Revised 29 March 2025
Accepted 31 March 2025

KEYWORDS
Dielectric properties; penetration depth; physicochemical properties; pine nuts; sunflower seeds

Introduction

Edible seeds and nuts are a group of foods commonly consumed for their rich content of proteins (higher than 20% w.b.), vitamins, minerals, and fatty acids, which also exhibit antioxidant properties due to their bioactive compounds.[1,2] Bioactive compounds are secondary metabolites from plant materials whose exhibit antioxidant activity with valuable and positive effects on human health. Among the edible seeds traditionally consumed worldwide are sunflower (*Helianthus annuus* L.), macadamia nuts (*Macadamia integrifolia*), almonds (*Prunus dulcis*), pine nut (*Pinus pinea* L.), and walnut (*Juglans regia* L.).[1–3]

Sunflower seed is a native oilseed of North America. It is among the most important oilseed crops because of its oil content, which had polyunsaturated fatty acids.[4,5] Sunflower seeds are made of around 8% water, 6% dietary fiber, 19% carbohydrates, 20% protein, and 48% crude fat,[6] almost

CONTACT María Elena Sosa-Morales msosa@ugto.mx Departamento de Alimentos, División de Ciencias de la Vida, Campus Irapuato-Salamanca, Universidad de Guanajuato, Carretera Irapuato-Silao km. 9, Irapuato 36500, Mexico

a third part of the fat content consists of essential fatty acids, including linoleic acid.[7] The composition of sunflower seeds has been reviewed by Zhang et al.[5] highlighting important benefits for health.

The pine nut is one of the nine main nut species worldwide and holds significant economic importance, primarily growing in Mediterranean countries.[8] Pine nuts are composed by unsaturated fatty acids, proteins, vitamins, minerals, and bioactive compounds.[9] The oil extracted from pine nuts is known for its bioactive compounds and antioxidant properties due to its high levels of phenols and vitamins.[10] Regular consumption of pine oil has been associated with various health benefits, such as reduction of inflammation, regulation of immune disorders, appetite control, and reducing blood fat levels.[10,11]

However, sunflower seeds and pine nuts, when exposed to high relative humidity during storage, are prone to fungal attacks that can lead to the development of aflatoxins.[12] Additionally, their high oil content makes them susceptible to oxidative reactions, such as rancidity, which reduces the nutritional value of foods and produces compounds that generate undesirable odors and flavors.[8,13] Both aflatoxins and rancidity products can negatively impact human health due to their mutagenic and carcinogenic effects.[12,13] Radio frequency and microwave treatments have gained attention in research due to their diverse applications in disinfection, pest management, enzyme inactivation, and processes like drying, roasting, and pasteurization.[14] Furthermore, understanding dielectric properties is essential for designing treatments that use electromagnetic energy, as they describe the interaction between electromagnetic energy and food.[15] Although several studies have been conducted on the dielectric properties of edible seeds such as almonds,[16] walnuts,[17] and peanuts,[18] there is not much information available in the literature on the dielectric properties of sunflower seeds and pine nuts. Therefore, this research aimed to study the dielectric properties of in-shell and shelled sunflower seeds and pine nuts at different frequencies and temperatures. Additionally, this study examines two types of permittivity: bulk permittivity, representing the air-sample mixture, and particle permittivity, which excludes air and reflects the permittivity of the sample as a solid.

Materials and methods

Materials

In-shell and shelled samples of sunflower seeds (*Helianthus annuus*) and pine nuts (*Pinus pinea*) were purchased from a local market (Central Supply of the Municipality, Irapuato, Guanajuato, Mexico). They were stored in plastic bags at 15°C and no pre-treatment was applied to them.

Methods

Characterization of in-shell and shelled sunflower seeds and pine nuts

The sunflower seeds and pine nuts were characterized by their moisture content, water activity, fat content, color, densities (bulk, tapped, particle), and porosity. The analyses were performed in triplicate for both in-shell and shelled samples. Moisture content was determined following the AOAC gravimetric method,[19] 2.0 ± 0.001 g of each sample were dried at 103 ± 2°C for 20 h in a convection oven (CE3F, Shel Lab, Cornelius OR, USA). Water activity (a_w) was measured with a dew point hygrometer (AquaLab Series 3, Decagon Devices, Pullman WA, USA) using 5.0 ± 0.01 g. Diethyl ether extraction was used to determine the fat content using a Goldfish equipment (GF-6, Novatech, Mexico City, CDMX, Mexico) during 5 h, with a sample of 2.0 ± 0.001 g.[19] Color parameters ($L^*a^*b^*$) of a 20 ± 0.01 g sample were analyzed with a colorimeter (ColorFlex EZ, Hunter Lab, Reston VA, USA), pre-calibrated with a black glass and a white tile. The bulk (ρ_{bulk}) and tapped (ρ_{tap}) densities were determined with the modified method by Hernández-Nava et al.[20] The ρ_{bulk} was measured by recording the volume of in 5 ± 0.01 g of the sample inside of a 50-mL graduated cylinder. Likewise, ρ_{tap} was determined when the volume of the sample remained constant after being manually compressed with a metallic tip. The particle density (ρ_{part}) was obtained following method described by Kaur et al.,[21] with slight modifications. A sample of 5 ± 0.01 g was placed in a 50 mL graduated cylinder, followed by the addition of 15 mL of acetone, and the volume displacement was recorded. The samples' porosity was calculated using the relationship between the bulk and particle densities according to Equation (1)[22]:

$$Porosity = 1 - \frac{\rho_{bulk}}{\rho_{part}} \quad (1)$$

Dielectric properties

A modified version of the Thru-Reflect-Line method was used to measure the dielectric as described by Engen and Hoer,[23] where two lines of similar impedance and different length are required. In this method, measurement with both lines is required to obtain the matrix T according to Equation (2) and (3).

$$M_1 = T_A T_{L1} T_B \quad (2)$$

$$M_2 = T_A T_{L2} T_B \quad (3)$$

where M_1 and M_2 are the measured T parameters from the sample, T_A and T_B are the T parameters of the connectors, which are equal for both lines, and T_{L1} and T_{L2} are the T parameters of the lines. The line T parameters are directly related to their propagation constant, and length as shown in Equation (4) and (5).

$$\mathrm{T}T_L = T_\Gamma T_{50L} {T_\Gamma}^{-1} \quad (4)$$

$$T_{50L} = \begin{pmatrix} e^{-\Upsilon l} & 0 \\ 0 & e^{-\Upsilon l} \end{pmatrix} \quad (5)$$

where T_Γ is the line reflection matrix and Υ is the complex propagation constant of the lines, and according to Equation (6):

$$\Upsilon = \alpha + j\beta \quad (6)$$

where α is the attenuation constant and β is the phase constant. The latter one is related to the effective permittivity of the line as shown in Equation (7):

$$\varepsilon_{eff} = \left(\frac{\beta c}{2\pi f}\right)^2 \quad (7)$$

where β is the phase constant, c is the speed of light in free space (3×10^8 m/s), and f is the frequency (Hz).

The Thru-Reflect-Line method involved constructing two lines; however, to simplify the process, only one line was physically built, while the second was simulated using a full-wave electromagnetic simulator (High Frequency Structure Simulation software, HFSS v7, ANSYS Inc., Canonsburg PA, USA). The sensor consisted of a microstrip transmission line designed with three layers. The top and bottom layers were composed of a TMM6 substrate with a relative permittivity of $\varepsilon r = 6$ and a thickness of h = 0.76 mm. The microstrip line was etched on the top layer, and the bottom layer functioned as the ground plane. The middle layer, which acted as a sample holder, was fabricated from PLA material using a 3D printer (Finder2, FlashForge Ltd., ZhengJiang, China) as shown in Figure 1.

In microstrip lines, for good transmission, it is important that the dielectric height is less than the wavelength ($H < \lambda$); this ensures a quasi-TEM transmission mode. Although perfect transmission is not required, maintaining the propagation mode is essential. Through electromagnetic simulation, it was found that a maximum height of 3.5 mm preserved the line's characteristics,

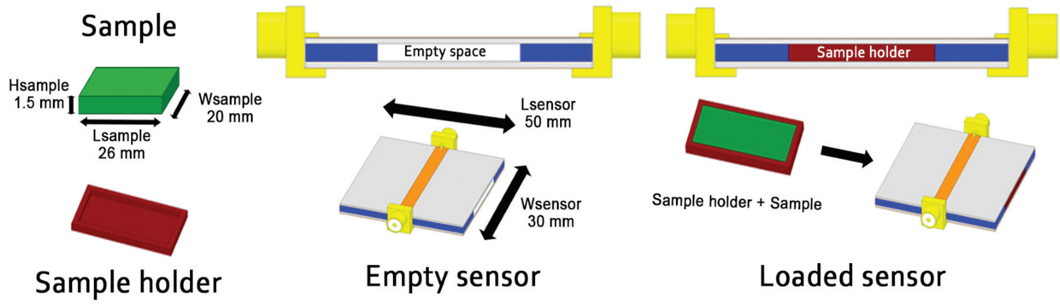

Figure 1. Sensor design used in the modified Thru-Reflect-Line method.

allowing the method to be applied up to 5 GHz. For this reason, the sample holder was designed to have $H_{sample} = 1.5$ mm. Moreover, if whole samples were used, due to the large seed size, big air gaps compared to the size of the transmission line would fill spaces between seeds; therefore, the preferred method for measuring their dielectric properties would be cavity method or transmission line method.[24,25]

Calibration was carried out using five substrates with known properties (DiClad880, RO4003, FR4, TMM6, TMM10i, purchased to Rogers Corporation (Chandler AZ, USA); calibration was done once at the beginning of every experimental run. Then, the Thru-Reflect-Line method was applied to both the physical measurement and the simulation. Each measurement was repeated five times, and a calibration curve was generated through curve fitting.

Measurement of the dielectric properties

All samples were ground using a commercial grinder (Model 80393, Hamilton Beach, Glen Allen VA, USA). Then, 2.0 ± 0.01 g of the final powder was filled inside the sample holder and pressed to avoid air gaps. All measurements were taken with a frequency range from 27 to 5000 MHz at three temperatures (20, 40 and 60°C), the samples were heated up using a commercial microwave oven (IOIO110MDI, Mabe, Querétaro, Mexico). The temperature was measured using an infrared thermometer (GM550, Fei Niao, Beijing, China) at different spots on the sample. Based on the measurements and because of the small size of sample (2 g), we get temperature uniformity. Then, their dielectric properties were measured immediately. All results were measured in quintuplicate. Every time that the sample was placed, it was tapped to avoid air. Since the experimental values consist of a sample/air mixture, where air lowers the measured permittivity, the solid sample permittivity (particle permittivity) was calculated using Equation (8).[26]

$$\varepsilon_{part} = \left[\frac{{\varepsilon_{Bulk}}^{1/3} + v_s - 1}{v_s}\right]^3 \tag{8}$$

where ε_{part} is the particle permittivity, ε_{bulk} is the bulk permittivity (air/sample mixture), and $v_s = \rho_{tap}/\rho_{part}$.

Penetration depth

The penetration depth (d_p) is defined as the volume where the microwave power density drops to *1/e* (e = 2.718). Since food materials are usually nonmagnetic, Metaxas and Meredith's model (Equation (9))[27] can be used to calculate the *dp*:

$$dp = \frac{c}{2\pi f\sqrt{2\varepsilon'\left[\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right]}} \tag{9}$$

where c is the speed of light in free space (3×10^8 m/s), f is the frequency (Hz), and ε' and ε'' the values of the dielectric constant and loss factor, respectively.

Statistical analysis

The data were analyzed statistically using analysis of variance (ANOVA) and Fisher's comparison tests at a 95% confidence level, performed with Minitab software (version 17, Minitab Inc., State College PA, USA). Using the same statistical software, the Pearson correlation coefficient (r) was calculated to analyze the effect of frequency and temperature on dielectric properties at a 95% confidence level.

Results and discussion

Physicochemical properties

The physicochemical properties of in-shell sunflower seed (ISS), shelled sunflower seed (SSS), in-shell pine nut (IPN), and shelled pine nut (SPN) are shown in Table 1. It was observed that, for sunflower and pine nuts, the in-shell samples had higher moisture content values ($p < .05$) due to the lignin and cellulolytic content in the shell that protect the kernels.[28–30] In the case of IPN, the difference in the moisture content observed (9.60 ± 0.12% w.b.) is due to several factors such as freshness, storage and harvesting conditions.[31,32] Regarding water activity (a_w), the sample with the lowest value was SPN (0.385 ± 0.002). The rest of the samples had a_w values higher than 0.5 ($p < .05$). a_w values above 0.5 might favor the lipid oxidation, potentially reducing the nutritional value and generating undesirable flavors and odors.[8,13] For fat content, the shelled samples had higher values than the in-shell ones (Table 1). The lower fat content values observed ($p < .05$) for in-shell samples is explained since shells are primarily composed of lignin and cellulolytic materials, which can account for around 80% of the seed's total weight.[28] The color parameters (L^*, a^*, b^*) values had a significant difference ($p < .05$) between samples. The L^* values of the in-shell samples were below 50 representing dark colors.[33,34] All the samples had positive values of a^* and b^*, with a tendency toward red and yellow, respectively.[33,35] The in-shell samples had higher a* values, indicating a redder shade than shelled ones. As for b^* values, shelled samples had higher values than the in-shell ones, which means a more yellow shade. The increase in L^* and the decrease of a^* values in the shelled samples are due to the removal of phenolic compounds such as flavonoids, anthocyanins, and tannins present in the seed shells of sunflowers and pine nuts.[29,30] The increase in b^* values in the shelled samples is the result of the prevalence of carotenoids, which give the characteristic colors of sunflower and pine nut kernels.[30,35] For bulk density (ρ_{bulk}) it was observed that in sunflower samples, the values slightly increased when the shell was removed ($p < .05$); meanwhile, in pine nut samples, the opposite behavior was observed ($p < .05$). The discrepancy in the behavior of sunflower seeds and pine nuts could be related to the characteristics of the cellular structure in in-shell seeds, in-shell nuts, and their kernels,

Table 1. Physicochemical properties of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.).

Samples	ISS	SSS	IPN	SPN
Moisture content (% w.b.)	6.59 ± 0.13^a	4.46 ± 0.09^b	9.60 ± 0.12^c	2.37 ± 0.23^d
a_w (25°C)	0.527 ± 0.004^a	0.548 ± 0.004^b	0.616 ± 0.003^c	0.385 ± 0.002^d
Fat content (% w.b.)	26.44 ± 1.67^a	47.50 ± 1.83^b	21.27 ± 1.58^c	50.59 ± 1.75^b
L^*	44.80 ± 0.95^a	54.43 ± 0.99^b	37.57 ± 0.15^c	60.87 ± 0.82^d
a^*	2.65 ± 0.10^a	3.76 ± 0.25^b	10.92 ± 0.44^c	5.53 ± 0.20^d
b^*	10.61 ± 0.35^a	16.15 ± 0.52^b	21.67 ± 0.61^c	28.47 ± 0.33^d
ρ_{bulk} (g/cm^3)	0.341 ± 0.010^a	0.464 ± 0.012^b	0.601 ± 0.007^c	0.518 ± 0.018^d
ρ_{tap} (g/cm^3)	0.390 ± 0.013^a	0.587 ± 0.008^b	0.637 ± 0.009^b	0.924 ± 0.118^c
ρ_{part} (g/cm^3)	0.764 ± 0.050^a	1.048 ± 0.045^b	1.135 ± 0.088^b	0.980 ± 0.039^b
Porosity	0.553 ± 0.017^a	0.557 ± 0.008^a	0.469 ± 0.035^b	0.472 ± 0.003^b

ISS: In-shell sunflower seed; SSS: Shelled sunflower seed; IPN: In-shell pine nut; SPN: Shelled pine nut.
Different letters in row indicate significant differences ($p < .05$) between samples according to ANOVA and Fisher's comparison test.

which influence the increase in mass and volume.[36] Tapped density (ρ_{tap}) and particle density (ρ_{part}) values were higher than those of ρ_{bulk}. Since smaller particles occupy the voids between larger particles due to the tapping process, a more compact packing arrangement results in higher ρ_{tap} values.[20,37] Similarly, ρ_{part} values are greater than ρ_{bulk} values due to the presence of air in ρ_{bulk}, which lowers its overall value.[38,39] Regarding porosity, the removal of the shell showed no significant difference ($p > .05$) in the porosity values of sunflower seed and pine nut samples. Furthermore, the sunflower seed samples had higher values than those of pine nuts ($p < .05$). Regarding porosity, a slight increase in the porosity values was observed with the removal of the shell, showing no significant difference ($p > .05$) in sunflower seed and pine nut samples. Furthermore, the sunflower seed samples had higher values than those of pine nuts ($p < .05$), suggesting a higher number of interparticle spaces. A larger number of spaces between particles indicates an increased amount of oxygen available for degradation reactions,[40] along with a decrease in dielectric properties caused by the presence of air.[26]

Factors influencing the dielectric constant using bulk permittivity

Frequency

The dielectric constant (ε_{bulk}') values, determined using the bulk permittivity, for in-shell and shelled sunflower seeds and pine nuts were significantly affected by frequency ($p < .05$). ε_{bulk}' decreased as the frequency increased, regardless of the temperature (Figure 2). For the Pearson correlation ($p < .05$), the average values of the samples when frequency increased at a fixed temperature were as follows: ISS (−0.72), SSS (−0.70), IPN (−0.69), and SPN (−0.67). As frequency increases, the ε_{bulk}' tends to decrease; however, the correlation is not extremely strong, as the values indicate a moderate to strong negative linear relationship. This effect was more pronounced in the radio frequency band than in the microwave band (Table 2). For example, at 20°C, when the frequency increased from 27 to 44 MHz, the ε_{bulk}' of ISS decreased from 3.04 to 2.47. In contrast, when the frequency increased from 915 to 5000 MHz, the ε_{bulk}' decreased from 1.49 to 1.39. The higher dielectric constant is attributed to interfacial and dipolar polarization. At low frequencies, this polarization follows the alternations of the electric field without lag, resulting in a higher dielectric constant. However, as the frequency increases, the dipoles are not able to follow the rapid changes in the polarity of the field, causing their oscillations to lag, which leads to a decrease in the dielectric constant.[41,42]

Temperature

When the temperature increased at a fixed frequency, the ε_{bulk}" decreased for in-shell and shelled sunflower seeds ($p < .05$). In contrast, the ε_{bulk}' increased for in-shell and shelled pine nuts. ISS and SSS had r values ($p < .05$) of −0.98 and −0.74, respectively, as the temperature rose while maintaining a constant frequency. This strong negative correlation could be due to thermal depolarization, where an increase in temperature disrupts dipole alignment, leading to a lower ε_{bulk}.'[43,44] In the case of IPN and SSS, the r values ($p < .05$) were 0.96 and 0.91, respectively, indicating a strong correlation where the ε_{bulk}' increases as temperature rises. This positive correlation could be because, in some materials, increasing temperature enhances ionic mobility or space charge polarization, leading to a higher dielectric constant.[15] The dielectric constant's behavior with temperature is complex, as it can either increase or decrease depending on the specific material.[43,44] This variable behavior can be explained in terms of a_w. As a_w increases with rising temperature, it influences how water availability and reactivity affect the dielectric properties.[45] For ISS and SSS, the ε_{bulk}' decreased with the increment of a_w due to water evaporation caused by moisture migration toward the surface, driven by pressure-induced flow.[45–47] In the cases of IPN and SPN, the ε_{bulk}" increased as a_w augmented, resulting from improved ion mobility due to more availability of free water molecules.[15,48]

Moisture and fat content

Moisture and fat affect the dielectric constant, with free water increasing its value, while fat tends to decrease it.[42,43] In the case of sunflower seeds, as the temperature increased at a fixed frequency, the

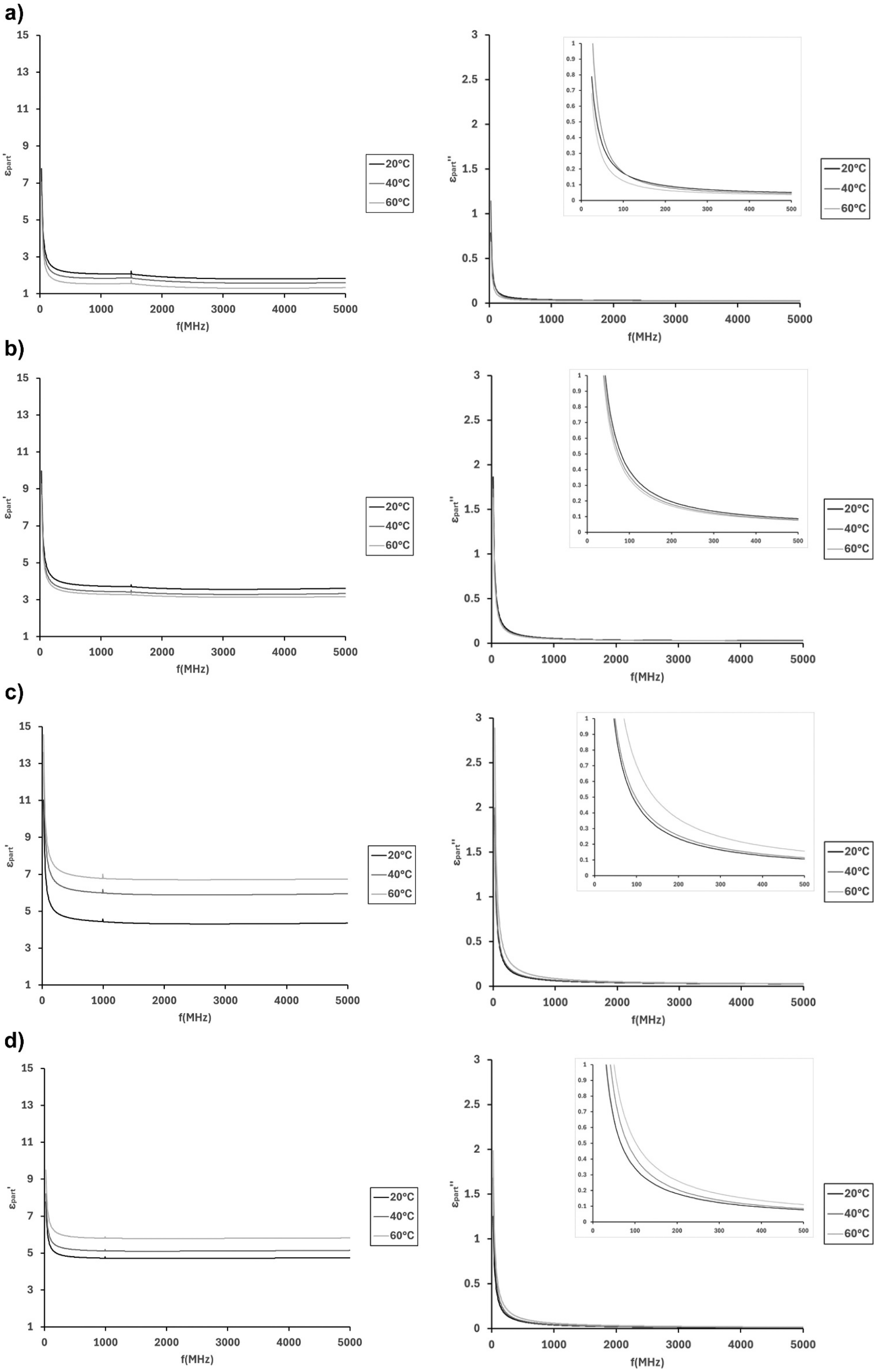

Figure 2. Dielectric constant (ε_{bulk}') and loss factor (ε_{bulk}'') using the bulk permittivity (air/sample mix) of in-shell sunflower seed (a), shelled sunflower seed (b), in-shell pine nut (c), and shelled pine nut (d).

Table 2. Dielectric properties of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.) using the bulk permittivity (air/sample mix).

		Dielectric constant (ε_{bulk}')					Loss factor (ε_{bulk}'')				
		Frequency (MHz)					Frequency (MHz)				
Sample	Temperature (°C)	27	44	915	2400	5000	27	44	915	2400	5000
ISS	20	$3.04 \pm 0.03^{A,a}$	$2.47 \pm 0.02^{B,a}$	$1.49 \pm 0.01^{C,a}$	$1.40 \pm 0.01^{D,a}$	$1.39 \pm 0.01^{D,a}$	$0.19 \pm 0.02^{A,a}$	$0.13 \pm 0.01^{B,a}$	$0.02 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$
	40	$2.84 \pm 0.02^{A,b}$	$2.31 \pm 0.02^{B,b}$	$1.39 \pm 0.02^{C,b}$	$1.30 \pm 0.02^{D,b}$	$1.29 \pm 0.02^{D,b}$	$0.27 \pm 0.02^{A,b}$	$0.17 \pm 0.01^{B,b}$	$0.01 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,a}$
	60	$2.63 \pm 0.05^{A,c}$	$2.12 \pm 0.04^{B,c}$	$1.26 \pm 0.02^{C,c}$	$1.17 \pm 0.02^{D,c}$	$1.16 \pm 0.02^{D,c}$	$0.17 \pm 0.02^{A,a}$	$0.11 \pm 0.01^{B,c}$	$0.01 \pm 0.00^{C,c}$	$0.01 \pm 0.00^{C,c}$	$0.01 \pm 0.00^{C,b}$
SSS	20	$4.06 \pm 0.14^{A,a}$	$3.39 \pm 0.12^{B,a}$	$2.25 \pm 0.10^{C,a}$	$2.18 \pm 0.10^{C,a}$	$2.20 \pm 0.10^{C,a}$	$0.51 \pm 0.04^{A,a}$	$0.33 \pm 0.02^{B,a}$	$0.02 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$
	40	$3.85 \pm 0.12^{A,b}$	$3.22 \pm 0.10^{B,b}$	$2.13 \pm 0.07^{C,b}$	$2.07 \pm 0.07^{C,b}$	$2.08 \pm 0.07^{C,b}$	$0.47 \pm 0.03^{A,a,b}$	$0.31 \pm 0.02^{B,a,b}$	$0.02 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,b}$
	60	$3.68 \pm 0.12^{A,c}$	$3.09 \pm 0.10^{B,b}$	$2.07 \pm 0.07^{C,b}$	$2.01 \pm 0.07^{C,b}$	$2.01 \pm 0.07^{C,b}$	$0.45 \pm 0.02^{A,b}$	$0.30 \pm 0.01^{B,b}$	$0.02 \pm 0.00^{C,c}$	$0.01 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,a,b}$
IPN	20	$4.41 \pm 0.05^{A,a}$	$3.73 \pm 0.05^{B,a}$	$2.53 \pm 0.06^{C,a}$	$2.48 \pm 0.06^{C,a}$	$2.50 \pm 0.06^{C,a}$	$0.52 \pm 0.05^{A,a}$	$0.35 \pm 0.03^{B,a}$	$0.03 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,a}$
	40	$5.15 \pm 0.09^{A,b}$	$4.40 \pm 0.08^{B,b}$	$3.09 \pm 0.07^{C,b}$	$3.05 \pm 0.07^{C,b}$	$3.07 \pm 0.06^{C,b}$	$0.53 \pm 0.04^{A,a}$	$0.36 \pm 0.02^{B,a}$	$0.03 \pm 0.00^{C,a}$	$0.01 \pm 0.00^{C,b}$	$0.01 \pm 0.00^{C,b}$
	60	$5.48 \pm 0.15^{A,c}$	$4.72 \pm 0.14^{B,c}$	$3.40 \pm 0.13^{C,c}$	$3.37 \pm 0.13^{C,c}$	$3.38 \pm 0.13^{C,c}$	$0.75 \pm 0.05^{A,b}$	$0.51 \pm 0.03^{B,b}$	$0.03 \pm 0.00^{C,b}$	$0.02 \pm 0.00^{C,c}$	$0.01 \pm 0.00^{C,c}$
SPN	20	$6.62 \pm 0.13^{A,a}$	$5.80 \pm 0.11^{B,a}$	$4.40 \pm 0.08^{C,a}$	$4.39 \pm 0.09^{C,a}$	$4.42 \pm 0.09^{C,a}$	$0.94 \pm 0.04^{A,a}$	$0.64 \pm 0.02^{B,a}$	$0.04 \pm 0.00^{C,a}$	$0.02 \pm 0.00^{C,D,a}$	$0.01 \pm 0.00^{D,a}$
	40	$7.00 \pm 0.11^{A,b}$	$6.18 \pm 0.11^{B,b}$	$4.75 \pm 0.10^{C,b}$	$4.74 \pm 0.10^{C,b}$	$4.78 \pm 0.10^{C,b}$	$1.24 \pm 0.03^{A,b}$	$0.82 \pm 0.02^{B,b}$	$0.04 \pm 0.00^{C,b}$	$0.02 \pm 0.00^{D,b}$	$0.01 \pm 0.00^{D,b}$
	60	$8.02 \pm 0.33^{A,c}$	$7.03 \pm 0.32^{B,c}$	$5.37 \pm 0.30^{C,c}$	$5.36 \pm 0.30^{C,c}$	$5.38 \pm 0.30^{C,c}$	$1.47 \pm 0.11^{A,c}$	$0.99 \pm 0.06^{B,c}$	$0.06 \pm 0.00^{C,c}$	$0.03 \pm 0.00^{C,c}$	$0.02 \pm 0.00^{C,c}$

ISS: In-shell sunflower seed; SSS: Shelled sunflower seed; IPN: In-shell pine nut; SPN: Shelled pine nut.

Different lowercase letters in the column indicate significant differences ($p < .05$) between samples with the same characteristics at different temperatures according to ANOVA and Fisher's comparison test.

Different capital letters in row show significant differences ($p < .05$) between samples at different frequencies of the same dielectric property according to ANOVA and Fisher's comparison test.

ISS sample, with higher moisture content and lower fat content (Table 1), showed a greater decrease in the ε_{bulk}" than the SSS sample, which had a lower moisture content and higher fat content. For example, for the ISS sample, at a frequency of 27 MHz, as the temperature increased from 20 to 60°C, the ε_{bulk}" decreased by 0.41, compared to the SSS sample, where the value decreased by 0.38. On the contrary, for the pine nut samples, the sample with higher moisture content and lower fat content (IPN) showed a smaller increase in the ε_{bulk}' as the temperature increased at a fixed frequency, compared to the SPN sample, which had a lower moisture content and higher fat content. For example, when the temperature increased from 20 to 60°C at a frequency of 27 MHz, the increase in the IPN sample was 1.07, while for SPN, it was 1.4.

Density and porosity

Overall, ε_{bulk}' values for in-shell and shelled pine nuts were higher than those for in-shell and shelled sunflower seeds at a fixed temperature and frequency. The dependence of the dielectric constant on density can explain this difference. It has been reported that, in agricultural products, an increase in ρ_{bulk} also increases ε_{bulk}' ,higher ρ_{bulk} values indicate denser packing, which reduces porosity.[49,50] In the present study, pine nut samples exhibited higher ρ_{bulk} values than sunflower seed samples ($p < .05$), which corresponded to lower porosity values (Table 1), resulting in higher ε_{bulk}' values.

Values of dielectric constant for shelled sunflower seeds at 915 and 2400 MHz at 20°C (2.25 and 2.18, respectively) are close to the reported by Li et al.[16] for almonds at the same frequencies and temperature (1.8 and 1.55, respectively). Similarities are due to near moisture content between them (4.46% w.b. for in-shell sunflower seeds and 4.2% w.b. for almonds). Also, the average value of shelled sunflower seeds at 915 MHz, 20°C ($\varepsilon_{bulk}' = 2.25$) is similar to the reported for almonds (1.7) and walnuts (2.2) at same conditions, reported by Wang et al.[17] Both reports[16,17] employed the open-ended coaxial probe method, thus, the Thru-Reflect-Line method from our study shows equivalent data and applicability for the dielectric properties measurements in this kind of seeds.

Factors influencing the loss factor using bulk permittivity

Frequency

The loss factor (ε_{bulk}"), calculated with the bulk permittivity, decreased with increasing frequency (Figure 2). The average values of the samples for the Pearson correlation ($p < .05$), when the frequency increased at a fixed temperature, were as follows: ISS (−0.69), SSS (−0.70), IPN (−0.71), and SPN (−0.71). The correlation values indicate a moderate to strong negative relationship: as the frequency increases, the ε_{bulk}" decreases. This can be attributed to the material's inability to efficiently follow high-frequency oscillations, resulting in lower energy dissipation at higher frequencies.[41,42] This decreasing effect was more noticeable in the radio frequency band compared to the microwave band (Table 2). For instance, at 20°C, as the frequency increased from 27 to 44 MHz, the ε_{bulk}" of ISS dropped from 0.19 to 0.13. In contrast, when the frequency rose from 915 to 2400 MHz, the ε_{bulk}" decreased from 0.02 to 0.01. This phenomenon is linked to the microscopic response mechanism of dielectric behavior: in the radio frequency band, dielectric loss is primarily due to the movement of free charges (ions, electrons), while in the microwave band, it is mainly caused by the rotation of polar molecules under an electric field.

Temperature

In the case of temperature increase, similar to the dielectric constant, the loss factor of the sunflower samples tends to decrease. In contrast, the values for pine nut samples increase with rising temperature. Additionally, as the frequency increases, these decreases or increases become smaller. The average r value ($p < .05$), for the sunflower seed samples was −0.65, and for the pine nut samples, it was 0.77. These correlation values suggest a moderate negative relationship for the sunflower samples and a strong positive relationship for the pine nut samples. The decrease in the loss factor as temperature rises could indicate that conduction losses are decreasing, possibly because the ions lack a suitable solvation medium, reducing charge carrier mobility at higher temperatures.[51] This may be a result of water evaporation caused by the increase in water activity with rising temperature.[45–47] On the other hand, for the pine nut samples, the increase in temperature causes a decrease in the viscosity of biomaterials, which results in higher oscillation of the dipoles and increased ionic mobility, leading to an increase in the loss factor.[52,53]

Moisture and fat content

Similar to the dielectric constant, the loss factor is affected by the moisture and fat content present in food.[42,43] It was observed that, for both sunflower seed and pine nut samples, those with higher moisture content and lower fat content showed a lower decrease in the loss factor as the frequency increased. For example, at a temperature of 40°C and with a frequency increase from 27 to 5000 MHz, the ISS sample exhibited a decrease of 0.26, while the SSS sample showed a decrease of 0.46.

Density and porosity

Overall, the ε_{bulk}" values for pine nut samples were higher than those for sunflower seed samples at a fixed temperature and frequency. Similar to ε_{bulk},' ε_{bulk}" is dependent on density, where an increase in ρ_{bulk} also increases ε_{bulk}" due to denser packing and a reduction in porosity.[49,50]

Loss factor values for shelled sunflower seeds at 915 and 2400 MHz at 20°C were 0.02 and 0.01, equal to those reported for almonds by Li et al.[16] at same conditions. However, the ε_{bulk}" value for shelled sunflower seeds at 915 MHz (0.02) was lower than the reported by Wang et al.[17] for almonds and walnuts, found as 5.7 and 2.9, respectively.

Penetration depth

For penetration depth (d_p) values, calculated with ε_{bulk}' and ε_{bulk}", a constant decrease was observed with the increment of frequency (Table 3). Overall, the d_p values in the radio frequency band were higher than those in the microwave band for all samples. This can be explained by the more pronounced difference in the modification rates of $\varepsilon_{bulk'}$ and $\varepsilon_{bulk'}$ in the radio frequency band, whereas in the microwave band, this difference was smaller, affecting the behavior of d_p.[52] Greater penetration depths at lower frequencies promote uniform heating, while reduced penetration at higher frequencies primarily leads to surface heating.[15,44] Thus, radio frequency technology may be more

Table 3. Penetration depth of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.) at different frequencies and temperatures using the bulk permittivity (air/sample mix).

		Frequency (MHz)				
		27	44	915	2400	5000
Sample	Temperature (°C)	Penetration depth (m)				
ISS	20	16.03 ± 1.86^{A,a}	13.00 ± 1.22^{B,a}	4.04 ± 0.02^{C,a}	1.93 ± 0.01^{D,a}	1.02 ± 0.01^{E,a}
	40	10.91 ± 0.73^{A,b}	9.82 ± 0.49^{B,b}	4.36 ± 0.03^{C,b}	1.94 ± 0.02^{D,a}	0.98 ± 0.01^{E,b}
	60	16.86 ± 1.49^{A,c}	14.38 ± 1.02^{B,c}	4.41 ± 0.07^{C,c}	1.91 ± 0.02^{D,b}	0.96 ± 0.01^{E,c}
SSS	20	7.09 ± 0.40^{A,a}	6.04 ± 0.39^{B,a}	3.45 ± 0.10^{C,a}	2.08 ± 0.06^{D,a}	1.23 ± 0.04^{E,a}
	40	7.36 ± 0.47^{A,b}	6.29 ± 0.36^{B,b}	3.57 ± 0.07^{C,b}	2.11 ± 0.04^{D,b}	1.23 ± 0.03^{E,a}
	60	7.52 ± 0.40^{A,b}	6.46 ± 0.29^{B,b}	3.63 ± 0.10^{C,c}	2.10 ± 0.05^{D,a,b}	1.20 ± 0.03^{E,b}
IPN	20	7.19 ± 0.62^{A,a}	5.46 ± 0.44^{B,a}	3.21 ± 0.05^{C,a}	2.17 ± 0.03^{D,a}	1.41 ± 0.02^{E,a}
	40	7.67 ± 0.48^{A,b}	6.36 ± 0.34^{B,b}	3.52 ± 0.08^{C,b}	2.46 ± 0.04^{D,b}	1.65 ± 0.03^{E,b}
	60	5.54 ± 0.33^{A,c}	4.63 ± 0.26^{B,c}	2.91 ± 0.09^{C,c}	2.27 ± 0.06^{D,c}	1.68 ± 0.04^{E,c}
SPN	20	4.86 ± 0.19^{A,a}	4.07 ± 0.14^{B,a}	2.73 ± 0.05^{C,a}	2.43 ± 0.05^{D,a}	2.14 ± 0.07^{E,a}
	40	3.79 ± 0.09^{A,b}	3.28 ± 0.07^{B,b}	2.63 ± 0.05^{C,b}	2.26 ± 0.04^{D,b}	1.81 ± 0.04^{E,b}
	60	3.43 ± 0.22^{A,c}	2.92 ± 0.18^{B,c}	2.10 ± 0.09^{C,c}	1.77 ± 0.06^{D,c}	1.41 ± 0.04^{E,c}

ISS: In-shell sunflower seed; SSS: Shelled sunflower seed; IPN: In-shell pine nut; SPN: Shelled pine nut.
Different lowercase letters in the column indicate significant differences ($p < .05$) between samples with the same characteristics at different temperatures according to ANOVA and Fisher's comparison test.
Different capital letters in row show significant differences ($p < .05$) between samples at different frequencies according to ANOVA and Fisher's comparison test.

suitable for heating agricultural products due to its better heating uniformity. On the other hand, at lower frequencies, an increase in temperature led to a rise in d_p values of sunflower samples, while the d_p of pine nut samples showed a tendency to decrease as the temperature rose. In contrast, at higher frequencies, the d_p of sunflower samples decreased with increasing temperature, while the d_p of pine nut samples exhibited different trends: IPN showed a tendency to increase as the temperature rose, whereas SPN maintained its tendency to decrease as temperature increased. This discrepancy in the behavior of sunflower and pine nut samples is due to the combined effect of temperature, frequency, and sample composition on $\varepsilon_{bulk'}$ and $\varepsilon_{bulk''}$, resulting in a characteristic tendency in d_p for each sample.

Modified values of the dielectric properties and d_p applying the particle permittivity

The plots showing the corrected values of the dielectric constant (ε_{part}') and loss factor (ε_{part}') for all samples, after applying the particle permittivity, are shown in Figure 3. ε_{part}' and ε_{part}" exhibited the same tendencies as ε_{bulk}' and ε_{bulk}" when increasing the frequency and temperature for all samples. Nevertheless, the effect of fat content in the pine nuts samples is more pronounced for ε_{part}' and ε_{part}" when applying the particle permittivity (Table 4). It was observed that the ε_{part}' and ε_{part}" values for IPN (fat content: 21.27 ± 1.58%) were higher than those for SPN (fat content: 50.59 ± 1.75). The presence of fat decreases the dielectric properties values because the increase in fat content dilutes the water ratio within the food system.[42,43] In contrast, the sunflower seed samples exhibited a behavior opposite to that of the pine nut samples regarding fat content and its effect on dielectric properties. Although fat content could serve as a means to predict the dielectric properties, it is not always accurate due to the complex interactions with other components, such as protein, that also affect the dielectric properties.[42,44] A higher protein content increases the dielectric properties of food products such as meat[54] and milk.[55] The shelled sunflower seeds are richer in protein than the in-shell sunflower seeds,[56,57] and thus their dielectric properties are modified by the interaction with proteins and fats, giving the samples their particular behavior. Overall, ε_{part}' and ε_{part}" were higher than ε_{bulk}' and ε_{bulk}" for all samples, resulting from the elimination of the air effect in the agricultural products.[21,49] The d_p values obtained with the particle permittivity were lower compared to those obtained with the bulk permittivity (Table 4). This decrease in d_p values is due to the combined increase in both the dielectric constant and the loss factor across all samples.[58] Furthermore, although the d_p values reported in the literature are typically on the order of centimeters, they are generally associated with foods that have high moisture content.[44] However, the d_p values of foods with low moisture content tend to be higher, on the order of meters, due to the small values of ε' and ε" resulting in very low loss, which increases d_p.[51,53,59] Higher d_p values for all samples at 40°C and 60°C were observed at 44 MHz for radio frequency and 915 MHz for microwaves. This information could be useful for developing heat treatments for these agricultural products using radio frequency or microwaves.

Conclusions

Moisture content was higher in in-shell samples, due to the lignin and cellulolytic materials in the shells. Water activity was in the range of 0.385–0.616. Fat content was higher in shelled samples, likely because of the lignin and cellulolytic materials present in the shells. The in-shell samples exhibited darker colors and a redder shade compared to the shelled ones, which was attributed to the presence of phenolic compounds. Tapped and particle densities were greater than bulk density values, resulting from the rearrangement of particles in the available space and the removal of air. Sunflower seed samples had higher porosity due to their lower bulk density. For all samples, the dielectric constant (ε') and loss factor (ε"') calculated using ε_{bulk} and ε_{part} decreased as frequency increased, with a more pronounced effect in the radio frequency range compared to the microwave band. As temperature increased, ε" decreased in sunflower seed samples but increased in pine nut samples, likely due to differences in water activity

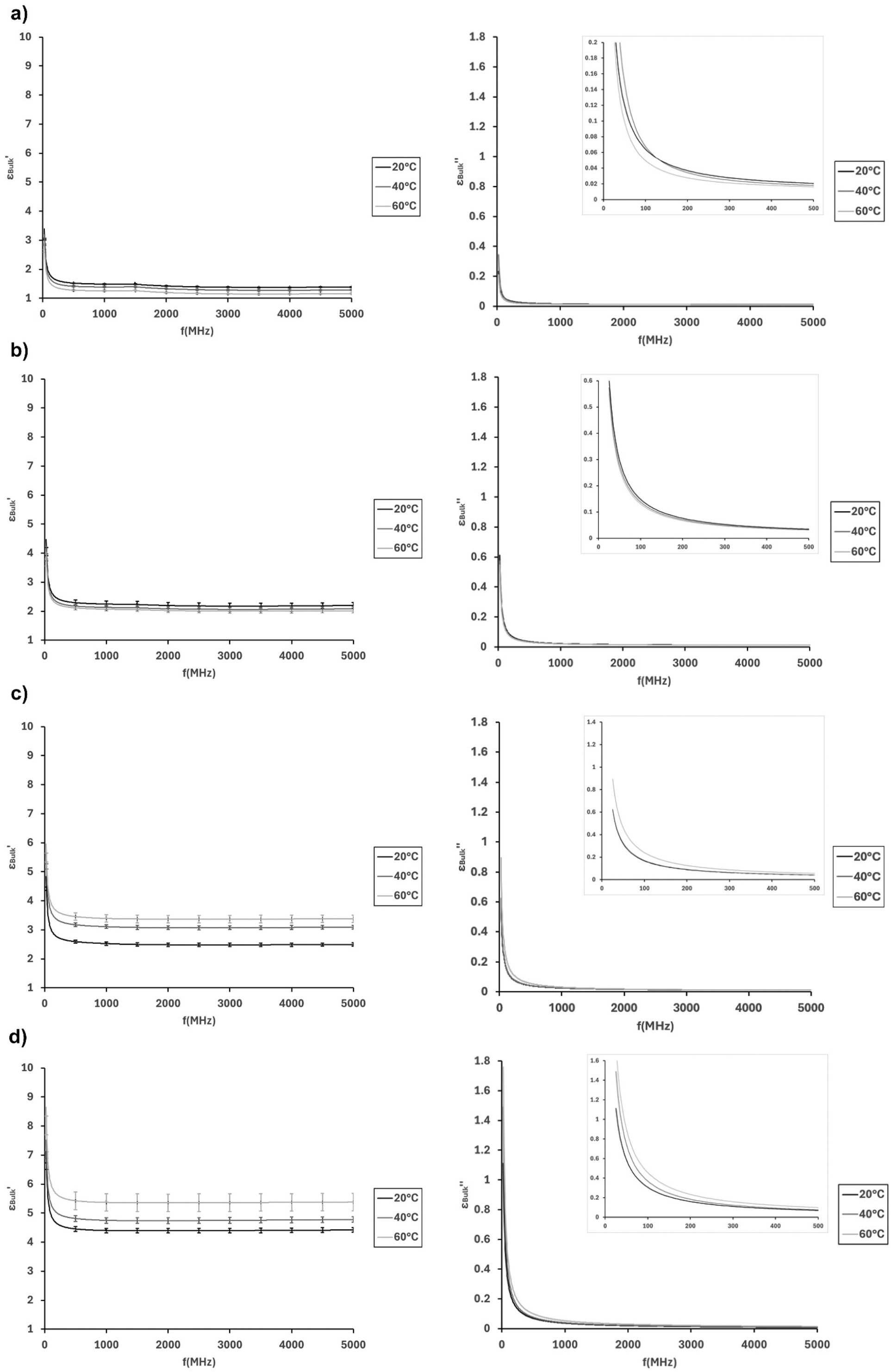

Figure 3. Dielectric constant (ε_{part}') and loss factor (ε_{part}'') using the particle permittivity (solid sample permittivity) of in-shell sunflower seed (a), shelled sunflower seed (b), in-shell pine nut (c), and shelled pine nut (d).

Table 4. Dielectric properties and penetration depth of in-shell and shelled sunflower seeds (*Helianthus annuus* L.) and pine nuts (*Pinus pinea* L.) at three temperatures and five frequencies using the particle permittivity (solid sample permittivity).

		Temperature (°C)								
		20			40			60		
Sample	Frequency (MHz)	ε_{part}'	ε_{part}''	d_p (m)	ε_{part}'	ε_{part}''	d_p (m)	ε_{part}'	ε_{part}''	d_p (m)
ISS	27	6.59	0.64	7.13	5.96	0.88	4.91	5.28	0.53	7.62
	44	4.79	0.40	5.91	4.30	0.50	4.52	3.76	0.32	6.66
	915	2.08	0.04	1.96	1.84	0.03	2.13	1.54	0.03	2.18
	2400	1.86	0.03	0.94	1.63	0.03	0.96	1.35	0.02	0.95
	5000	1.84	0.03	0.50	1.60	0.03	0.48	1.33	0.02	0.48
SSS	27	8.72	1.51	3.48	8.12	1.39	3.63	7.61	1.31	3.73
	44	6.81	0.94	3.01	6.33	0.87	3.14	5.96	0.82	3.24
	915	3.75	0.06	1.78	3.46	0.05	1.85	3.31	0.05	1.88
	2400	3.58	0.04	1.07	3.30	0.03	1.10	3.16	0.03	1.09
	5000	3.62	0.03	0.63	3.33	0.03	0.64	3.16	0.03	0.63
IPN	27	9.73	1.57	3.52	12.11	1.65	3.74	13.04	2.38	2.69
	44	7.74	1.02	2.95	9.80	1.08	3.14	10.68	1.57	2.27
	915	4.45	0.07	1.64	6.01	0.07	1.78	6.79	0.09	1.46
	2400	4.32	0.04	1.11	5.88	0.04	1.25	6.70	0.05	1.14
	5000	4.36	0.03	0.73	5.94	0.03	0.84	6.74	0.03	0.84
SPN	27	7.20	1.06	4.51	7.63	1.40	3.52	8.79	1.66	3.17
	44	6.28	0.72	3.78	6.71	0.92	3.05	7.67	1.12	2.70
	915	4.73	0.04	2.54	5.12	0.05	2.45	5.81	0.06	1.95
	2400	4.72	0.02	2.26	5.11	0.02	2.10	5.79	0.03	1.64
	5000	4.74	0.01	1.99	5.14	0.01	1.68	5.82	0.02	1.31

ISS: In-shell sunflower seed; SSS: Shelled sunflower seed; IPN: In-shell pine nut; SPN: Shelled pine nut.
Values are the average of analyses and calculations made by triplicate.

influencing evaporation and ion mobility. This effect was more evident in in-shell samples, which had higher moisture content and lower fat content. The loss factor (ε”) increased with rising temperature across all samples due to enhanced ionic mobility. Additionally, the dielectric properties were influenced by density, as an increase in ρ_{bulk} lead to higher ε_{bulk}‘ and ε_{bulk}” values, attributed to denser packing and reduced porosity. These different results among samples regarding ε’ and ε”, reflected the influence of density and food composition on dielectric behavior. Dielectric properties calculated with the particle permittivity (ε_{part}) were higher than those using bulk permittivity (ε_{bulk}), due to the removal of air. Penetration depth values were higher at lower frequencies, promoting uniform heating. The d_p values derived from ε_{part} were smaller compared to those calculated from ε_{bulk}, due to the simultaneous increase in the dielectric constant and loss factor. The information obtained in this study could be useful for developing heat treatments for sunflower seeds and pine nuts using radio frequency or microwave technologies applied in food processing and safety. Furthermore, it is suggested that future studies vary moisture content and water activity, as well as increase the temperature range to gather more information about the effects of these factors on the dielectric properties of these agricultural products.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This research was funded by Consejo Nacional de Humanidades, Ciencias y Tecnologías (CONAHCYT, now SECIHTI), research project [CBF2023-2024-1220].

ORCID

María Elena Sosa-Morales http://orcid.org/0000-0002-1197-2572

Author contributions

R. Hernández-Nava performed the physicochemical experiments and wrote the draft of the manuscript. D. Sarmiento-Narváez conducted experiments focused on dielectric properties. J.M. Meza-Arenas designed the sensor for measuring dielectric properties and analyzed the data. T. Kaur and A. Corona-Chávez assisted in interpreting the results and reviewing the manuscript. R. Rojas-Laguna performed the experiments for densities and ran the statistical analysis. M.E. Sosa-Morales designed the study, performed experiments for density, reviewed the data, and revised the manuscript.

Data availability statement

The datasets generated for this study are available upon request.

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