

EFFECTS OF La_2O_3 AND Bi_2O_3 ADDITIONS ON THE MICROWAVE DIELECTRIC AND ANTENNA PROPERTIES OF $\text{SrBi}_2\text{Nb}_2\text{O}_9$ CERAMIC MATRIX

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Abstract. This work presents a comparison between the dielectric properties of the $\text{SrBi}_2\text{Nb}_2\text{O}_9$ (SBN) ceramic matrix and the individual additions of Bi_2O_3 and La_2O_3 , suggesting the material that would be most suitable for microwave applications. The SBN sample was synthesized by solid-state reaction, and the X-ray diffraction method (XRD) was used for the structural characterization of the materials analyzed in this study. Thermal stability of the samples was measured experimentally by studying the temperature coefficient of resonant frequency (τ_f) the results confirm that the individual additions of 15 wt% of Bi_2O_3 or La_2O_3 improved the thermostability of the composites. The Hakki-Coleman method was used to obtain the dielectric properties in the microwave range, and it is possible to observe that the dielectric permittivity of the composites increased, while their loss tangents decreased. The numerical simulation demonstrated the functioning of the materials as dielectric resonating antennas (DRA), with a reflection coefficient below -10 dB in all samples.

Keywords: $\text{SrBi}_2\text{Nb}_2\text{O}_9$. Microwave. Bi_2O_3 . La_2O_3 . DRA.

1 Introduction

Dielectric ceramics have notable importance to the global communications system due to applications ranging from cellular telephony to global positioning systems (GPS). Their high permittivity and low losses allow the miniaturization and use of these materials in different electronic components, such as filters, oscillators, amplifiers, tuners, etc. (ABREU et al., 2021; MOULSON; HERBERT, 2003; SEBASTIAN, 2008; WANG et al., 2020).

SrBi₂Nb₂O₉ (SBN) ceramic matrix is a ferroelectric with structured layers of bismuth, has an Aurivillius phase, consisting of alternating layers of (SrNb₂O₂)²⁻ perovskite and (Bi₂O₂)²⁺. (KUMAR et al., 2019; WEI et al., 2020; WU, 2018). Ferroelectric materials have high permittiveness values, good piezoelectric coefficients, and spontaneous polarization, which make these materials suitable for many technological applications. In addition, these materials have high resistance to fatigue and their potential use in random access memories has been constantly investigated (SANCHO et al., 2013; SINGH et al., 2018; WU et al., 2017). Recent studies show the possibility of application as dielectric resonator antennas (DRA) of the SBN matrix with additions of Bi₂O₃ or La₂O₃ (ABREU et al., 2019; ABREU et al., 2020).

The objective of this work was to compare the influence of individual additions of 15 wt% of La₂O₃ or Bi₂O₃ on ceramic matrix SrBi₂Nb₂O₉ (SBN), showing the influence of these additions on the dielectric characteristics of this ceramic material and the possibility of applications in microwave devices (MW) of materials investigated. The effect of additions of the SBN pure phase on structural identification and its dielectric properties in the microwave range were analyzed. X-ray diffraction (XRD) was used for the structural characterization of the samples, and the confirmation of the phases was obtained through the Rietveld refinement (ABREU et al., 2021; NASCIMENTO et al., 2020; RIETVELD, 1969). Dielectric properties in the microwave region were studied using the Hakki-Coleman method (COURTNEY, 1970; HAKKI; COLEMAN, 1960), and through the technique developed by Long, MacAllister, and Shen (LONG; MCALLISTER; SHEN, 1983; MCALLISTER; LONG; CONWAY, 1983). Thermal stability of the material was investigated using the Silva-Fernandes-Sombra (SFS) method (SILVA; FERNANDES; SOMBRA, 2012). Numerical simulation was carried out to obtain the far-field parameters and, in this

way, to compare the behavior of the materials as a dielectric resonant antenna (DRA).

2 Materials and Methods

Samples were prepared through the conventional ceramic method. The precursor oxides SrCO₃, Bi₂O₃, Nb₂O₅ were ground for 8 h at 360 rpm using the Fritsch Pulverisette 5 mill, and calcined in a JUNG resistive oven (LF0912) at 900°C for 2h. After this process, 15 wt% of Bi₂O₃ or 15wt% of La₂O₃ was added on SBN, resulting in samples SBNBi and SBNLa. Samples were compacted via uniaxial pressure of 294 MPa in the bulk format using 5% glycerin as a binder and were sintered in the conventional controlled furnace (JUNG - LF0912), respectively, at 900°C / 2h, 850°C / 2h, and 1000°C / 2h.

Structural analysis of the samples was performed using X-ray diffraction (XRD), and the phases were identified using the X'Pert HighScore Plus program and the Inorganic Crystal Structure Database (ICSD).

Through the Hakki – Coleman method (COURTNEY, 1970; HAKKI; COLEMAN, 1960), and using the HP8716ET network analyzer from Hewlett - Packard (HP), the dielectric properties of the samples were obtained. The analysis of the sample's characteristic spectrum determines the resonance modes of the resonator, which allowed to calculate dielectric permittivity (ϵ_r), dielectric loss tangent ($\tan \delta$) and quality factor ($Q = 1 / \tan \delta$) of the compounds analyzed.

To analyze the thermal stability of the materials, the Silva-Fernandes-Sombra (SFS) method was employed. This method analyzes the dominant mode and τ_f can be calculated analytically using equation (1) (SILVA; FERNANDES; SOMBRA, 2012):

$$\tau_f = \frac{1}{f_0} \cdot \frac{\Delta f}{\Delta T} \cdot 10^6 \text{ ppm}/^\circ\text{C} \quad (1)$$

Where f_0 is the resonance frequency at the beginning of the process, Δf the difference between the final and initial frequencies, and ΔT the difference between the final and initial temperatures.

For the performance of the dielectric resonator antenna (DRA) was employed the technique developed by Long, Mcallister, and Shen using Agilent Network Analyzer N5230A network. The samples are excited under the DRA configuration (Fig. 1), the probe ($d = 9.6 \text{ mm}$) is connected to the ground plane made of copper ($35.5 \text{ cm} \times 30 \text{ cm} \times 2.14 \text{ mm}$). The cylindrical DRA

has a radius (a), height (h), and (d) is the height of the probe.

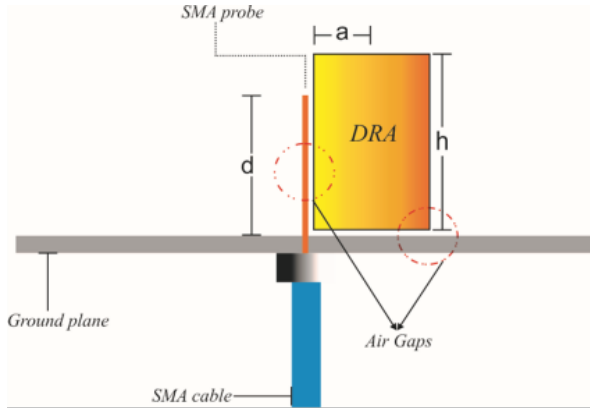


Figure 1: Setup for the DRA measurement

According to the literature (ABREU et al., 2021; ABREU et al., 2021), the quality factor Q and the resonant frequency can be accurately calculated according to the following equations,

$$Q_{(HE_{11\tau})} = 0.01007(\varepsilon_r^{1.3}) \cdot \frac{a}{h} \left[1 + 100e^{-2.05\left(\frac{a}{2h} - 0.0125\left(\frac{a}{h}\right)^2\right)} \right] \quad (2)$$

and,

$$f_{(HE_{11\tau})} = \frac{6.324c}{2\pi a\sqrt{\varepsilon_r+2}} \left[0.27 + 0.36\left(\frac{a}{2h}\right) + 0.02\left(\frac{a}{2h}\right)^2 \right] \quad (3)$$

From the experimental results of return loss (S11) and the real (Z') and imaginary (Z'') impedances, a numerical simulation was carried out to obtain far-field parameters, compare experimental results with simulated ones and, evaluate the application of materials such as DRA (HAYDOURA et al., 2021; OLIVEIRA et al., 2019). Ansoft HFSS® (High-Frequency Structure Simulator) software was used to perform the simulation and through it was possible to obtain the parameters: directivity, gain, antenna efficiency, and radiation diagram, etc (ABREU et al., 2021).

3 Results and discussion

SBN, SBNBi, and SBNLa phases were analyzed through X-ray diffraction. The diffractogram of the SBNBi, when compared to the SBN matrix (Fig. 2a), demonstrates the formation of a new phase that was identified as $(\text{Sr}_{0.6}\text{Bi}_{0.3}\text{O}_5)_2\text{Bi}_2\text{O}_7$ (JCPDS 73-923). For SBNLa samples (Fig. 2b) was observed the pre-

sence of LaNbO_4 (JCPDS n°. 81-618) as a secondary phase. When the added samples are compared to the SBN sample without addition, no significant shift in the peak positions was observed in the pure sample indicating that its crystalline structure was not affected by the additions.

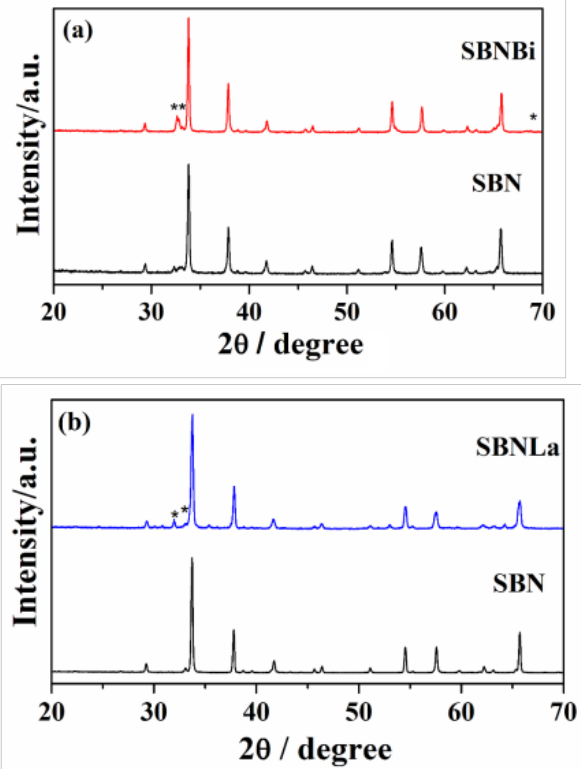


Figure 2: X-ray diffraction of SBN samples, (a) SBNBi and $\diamond \text{Bi}_3\text{NbO}_7$, (b) SBNLa, * LaNbO_4 .

In Fig. 3 a-c are shown results obtained by the Rietveld refinement of the samples, where SBN and $(\text{Sr}_{0.6}\text{Bi}_{0.3}\text{O}_5)_2\text{Bi}_2\text{O}_7$ phases were used in the refinement of the SBNBi, while SBN and LaNbO_4 were used to the refinement of the SBNLa. The low residual values between the observed and calculated diffraction patterns indicate that the refinement performed is reliable. The lattice parameters, convergence indexes Rwp (weighted residual error), Rexp (value expected for Rwp) and S (goodness of fit), where S is given by the Rwp / Rexp ratio, are shown in Table 1.

Through the Hakki – Coleman method (COURTNEY, 1970; HAKKI; COLEMAN, 1960), the dielectric properties ε_r , $\tan \delta$, and $Q_d \times f_r$ were obtained, and the results are shown in Table 2. Comparing the samples

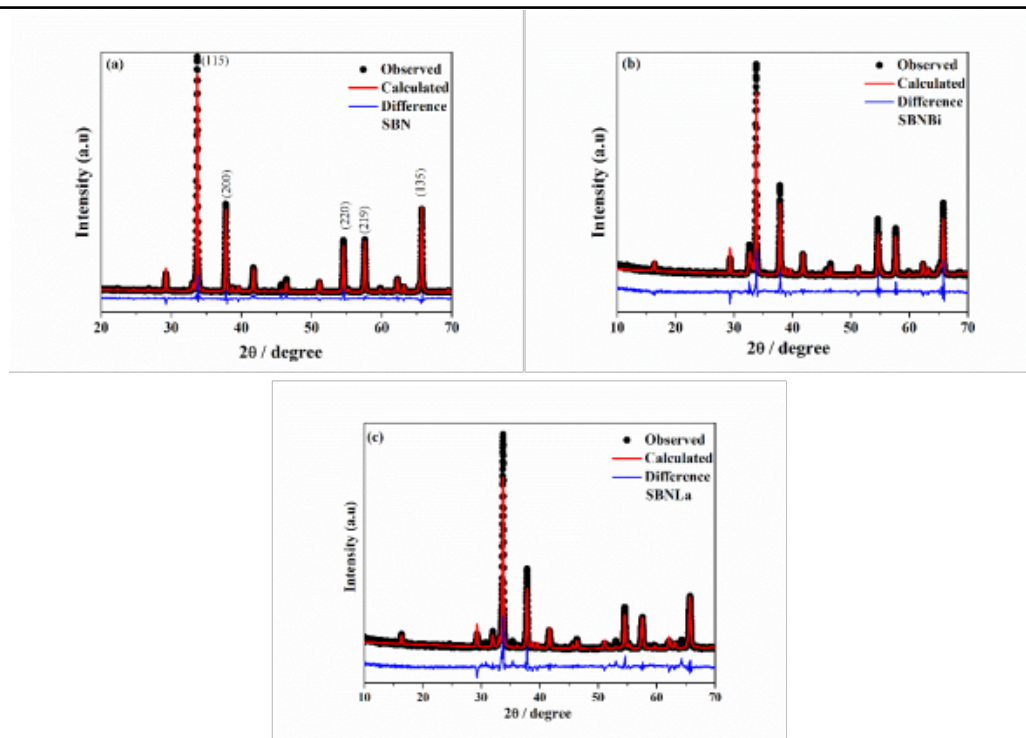


Figure 3: Rietveld refinement of samples from (a) SBN, (b) SBNBi, and (c) SBNLa.

Table 1: Parameters obtained in the Rietveld Refinement for SBN samples.

Samples	R-P (%)	R-WP (%)	S (%)	D-WP
SBN	12.43	17.19	1.90	0.13
SBNBi	17.43	22.07	1.61	0.06
SBNLa	17.78	22.76	1.75	0.05

added with the pure SBN, it was possible to notice that additions caused a decrease in the tangent of losses ($\tan \delta$), and the increase in electrical permittivity (ϵ_r) and the quality factor Q_d . The highest permittiveness ($\epsilon_r = 104.82$) and the lowest loss tangent ($\tan \delta = 0.0186$) were found for the SBNBi.

SBN pure phase presented a $\tau_f = -345.62$ ppm/°C, this fact was the initial motivation for the addition of oxides Bi_2O_3 or La_2O_3 , due to the possibility of obtaining new composites with higher thermal stability and better dielectric properties. SBNBi sample demonstrated $\tau_f = -235.02$ ppm/°C and SBNLa presented $\tau_f = -116.51$ ppm/°C, value closer to zero for addition with La_2O_3 could be explained by the formation of the LaNbO_4 phase that has positive τ_f (KIM et al., 2006). These results are extremely important for mi-

crowave applications, as they demonstrate an increase in the thermal stability of the SBN matrix and they encourage the manufacture of new composites with greater additions. Analyzing the results obtained, it is observed that the additions made in the SBN matrix caused the improvement of the main properties necessary for a DRA (ABREU et al., 2021; HAYDOURA et al., 2021; PETOSA, 2007).

Far-field parameters were obtained through numerical simulation using the Ansoft HFSS® software, these data provide the operation standards of the materials such as DRA. In the simulation performed, the experimental and simulated results for return loss (S_{11}), Z' , Z'' and the Smith Chart were compared. In Fig. 4 it is possible to observe the excellent fit between the experimental and simulated profile.

According to Table 3 and Fig. 4, it can be noted that all samples showed a return loss with values well below -10 dB, a value required for applications such as DRA (BALANIS, 2011). Return loss (S_{11}) indicates the loss of energy due to discontinuity in the transmission line/device, that is, the more negative this parameter is, the less energy is lost (POOLE; DARWAZEH, 2015). It is worth mentioning that the error between the

Table 2: Microwave measurements of materials using the Hakki-Coleman method.

Sample	D (mm)	H (mm)	ε_r	$\tan \delta$ (10^{-2})	$Q_d \times f_r$ (GHz)	τ_f (ppm/ $^{\circ}$ C)
SBN	17.78	8.96	28.6	3.24	135.37	-345.62
SBNBi	15.02	7.88	104.82	1.86	142.63	-235.02
SBNLa	16.94	8.40	23.54	1.87	273.44	-116.51

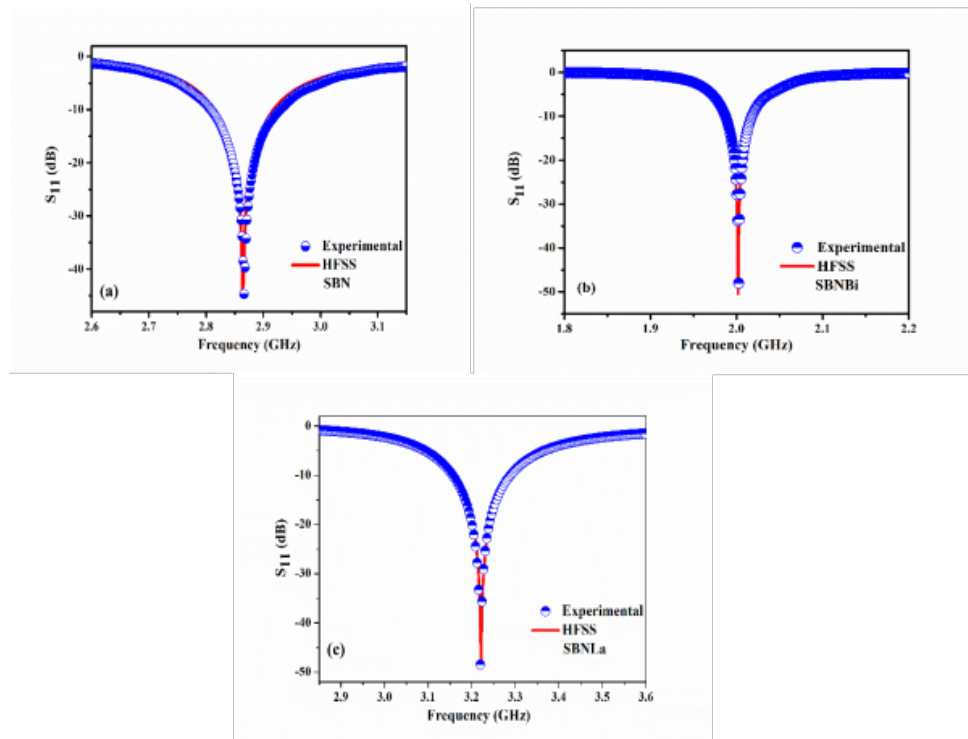


Figure 4: Return loss (S_{11}) of the samples (a) SBN, (b) SBNBi, (c) SBNLa.

simulated frequency and the measurement is practically zero for all samples, as can be seen in Table.

Table 3: Frequency and return loss (S_{11}) experimental and simulated for the $HE_{11\delta}$ resonant mode.

Sample	f_r (GHz)			S_{11} (dB)		
	Exp	Sim	Error (%)	Exp	Sim	Error (%)
SBN	2.86	2.86	0.00	-49.97	-45.01	0.08
SBNBi	2.86	2.86	0.00	-49.67	-48.03	3.41
SBNLa	3.22	3.21	0.06	-48.58	-48.49	0.22

Fig. 5 shows the experimental and simulated diagrams of the real Z' (Ω) and imaginary Z'' (Ω) of the complex impedance. It is possible to notice that all samples showed a good agreement indicating that the simulation performed was adequate.

Fig. 6 shows Smith Chart for the three samples analyzed in this study. It is possible to perceive an inductive character in SBN and SBNLa since its semi-circular regions are displaced upwards from the horizontal axis, while the SBNBi sample has a more capacitive character since its semi-circular regions are more displaced downwards horizontal axis (POOLE; DARWAZEH, 2015).

With the data obtained from the monopole experiment and using numerical simulation, it was possible to obtain the following far-field parameters: Directivity; Gain; Reflection coefficient (Γ); Voltage standing wave ratio (VSWR); Energy reflection coefficient (Γ_{pwr}); Energy transmission coefficient (T_{pwr}); Radiation efficiency (Eff). It is worth mentioning that these data provide the operation standards of materials such as DRA or other devices that operate in the microwave region (ABREU et al., 2021; POOLE; DARWAZEH, 2015).

Data from Table 4 demonstrate that the VSWR of all samples is very close to 1, indicating that a good part of the energy is being transmitted and little is lost by reflection. The energy transmission coefficient (T_{pwr}) indicates how much power is transmitted as a function of the incompatibility in the transmission line (POOLE; DARWAZEH, 2015), that is, the closer to 1 this value, the greater the energy transmission and the lower the loss due to impedance mismatch and all samples meet this requirement. The energy reflection coefficient (Γ_{pwr}) indicates how much energy is lost by reflection, the closer to zero this parameter is, the less energy is lost, and all samples have a value in the range of 10^{-5} . Finally, the radiation efficiency of the three samples was calculated, with the best efficiency being observed for

the SBNLa sample (Eff = 80.63%). The resonant frequency of the materials is in the range of 2 - 4 GHz allowing them to act as devices that operate in the S-band according to the classification from IEEE (BALANIS, 2012; CHANG, 2000; POOLE; DARWAZEH, 2015; POZAR, 2012; STUTZMAN; THIELE, 2012). The results obtained demonstrate that the simulation carried out suggests potential applications for the SBNBi and SBNLa phases as DRA's or in other devices that operate in the microwave range, such as microwave filters, oscillators, radar detectors, etc.

4 Conclusion

In this work, we have analyzed the effects of individual additions of Bi_2O_3 or La_2O_3 on $SrBi_2Nb_2O_9$ (SBN) ceramic matrix, comparing their dielectric properties as well as the performance of these materials as DRA. The structural analysis of the SBN matrix and additions was performed through X-ray diffraction (XRD), and the Rietveld refinement confirmed the presence of SBN, as well as the formation of secondary phases in the added samples. The analysis of the dielectric properties demonstrated that the additions caused the reduction of $\tan \delta$ and the increase of ϵ_r , in addition to causing an increase in the thermal stability of the SBN matrix.

Numerical simulation showed that all samples showed loss of return below -10 dB and operated in the region of the S-band (2 - 4 GHz), making them potential candidates for use in communication satellites and radars, and the results of the far-field parameters demonstrated that SBNLa demonstrated to be the most efficient material for application as DRA, since it presented higher values of directivity, gain and radiation efficiency.

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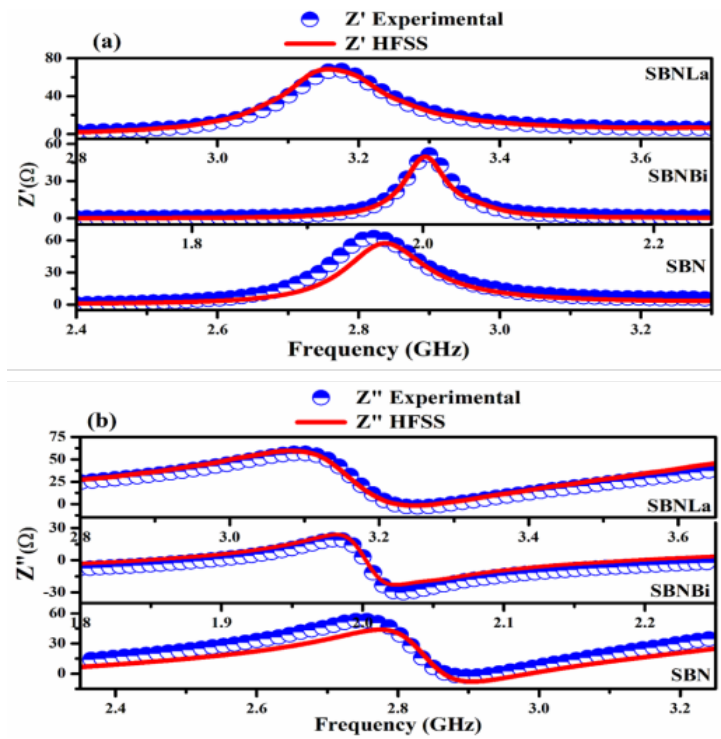


Figure 5: Experimental and simulated diagrams of the complex impedance (a) real part $Z'(\Omega)$ (b) imaginary part $Z''(\Omega)$.

Table 4: Directivity; Gain; Reflection coefficient (Γ); Voltage standing wave ratio (VSWR); Energy reflection coefficient (Γ_{pwr}); Energy transmission coefficient (T_{pwr}); Efficiency of radiation (Eff); Determined by the simulation in the HFSS.

Sample	Direc. (dBi)	Gain (dBi)	Γ	VSWR	Γ_{pwr}	T_{pwr}	Eff. (%)
SBN	6.62	5.56	0.003173	1.00636	0.00001	0.9999	78.19
SBNBi	6.90	3.11	0.003284	1.00659	0.00001	0.9999	41.85
SBNLa	6.92	5.98	0.003723	1.00747	0.00001	0.9999	80.63

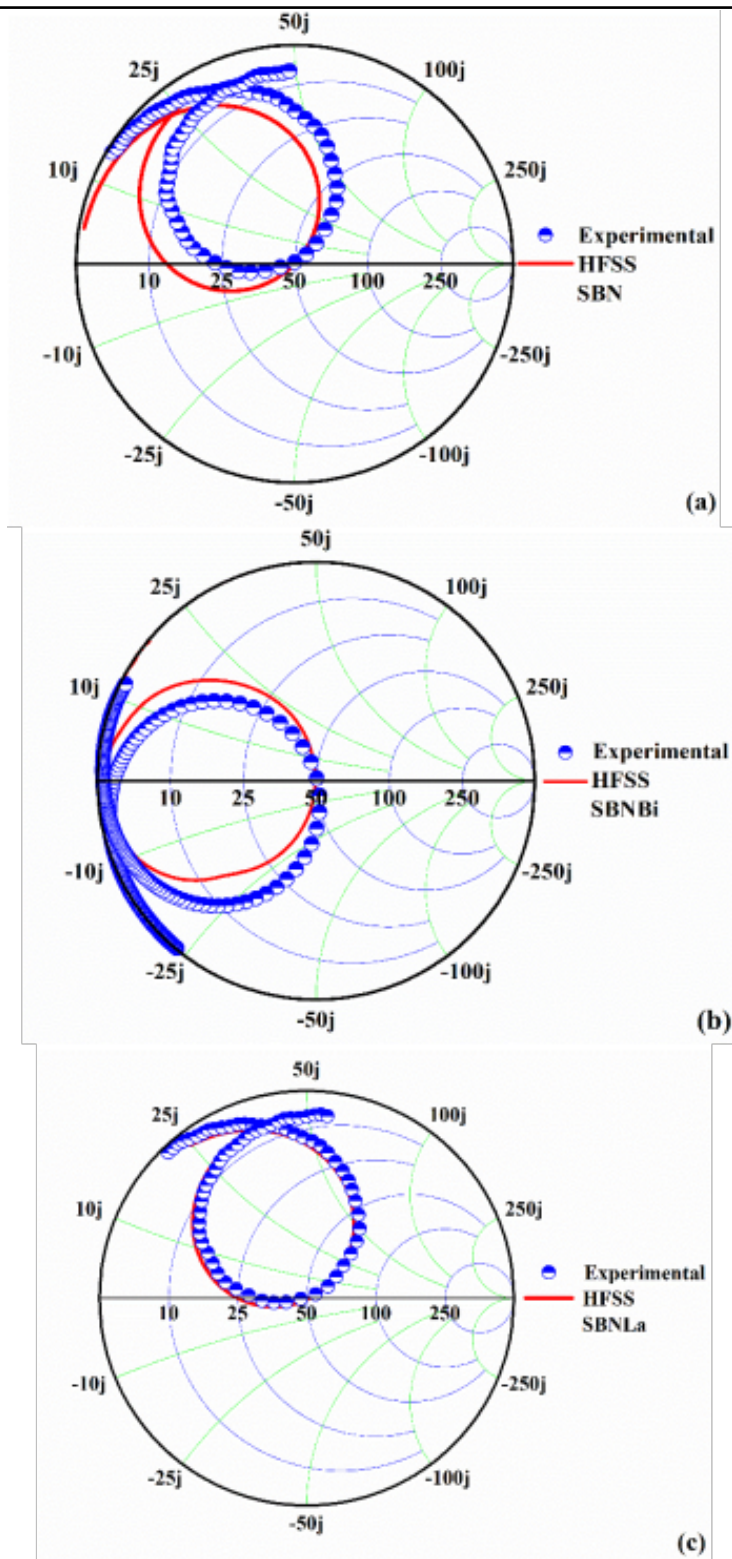


Figure 6: Smith charts experimental and simulated from: (a) SBN, (b) SBNB and (c) SBNLa.

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