Synthesis and Characterization of $Ni_{0.5}Co_{0.5}Tio_3$ Composite with Polyvinyl Alcohol (PVA) as Microwave Absorber

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Abstract. $Ni_{0,5}Co_{0,5}TiO_3$ composites are included in the category of magnetic materials with a perovskite structure that has great potential as a microwave absorbers due to their superior magnetic and dielectric properties, which are important for reducing electromagnetic interference (EMI). To improve the electromagnetic properties, $Ni_{0,5}Co_{0,5}TiO_3$ composites are mixed with conductive polymers such as Polyvinyl Alcohol (PVA). This research synthesizes $Ni_{0,5}Co_{0,5}TiO_3$ via solid-state mechanical milling techniques and characterizes its crystal structure, morphology, and microwave absorption capacity. The addition of PVA in this composite aims to increase the flexibility of the material while maintaining its absorption capacity. Based on the research conducted, the physical and electromagnetic properties of the composite are greatly influenced by the synthesis method, particle morphology, and cation distribution in crystal structure. This research focuses on the effect of the combination of $Ni_{0,5}Co_{0,5}TiO_3$ with PVA on microwave absorption efficiency, which is measured by the reflection loss value.

Keywords: $Ni_{0.5}Co_{0.5}TiO_3$, polyvinyl alcohol, microwave absorber, mechanical milling, material characterization.

1 Introduction

Radar Absorbing Material (RAM) was developed to reduce electromagnetic interference (EMI) that can cause circuit disturbances to system failure [1,2]. Materials based on $Ni_{0.5}Co_{0.5}TiO_3$**,** polyaniline (PAni)**,** TiO_2**,** and coconut shell carbon were used to enhance microwave absorption through energy conversion into heat [3,4]. Studies show that BaM/PET, BaM/PAni, and $MnFe_2O_4$ /PPy composites produce significant reflection loss at various frequencies [5,6]. This study developed a PAni/TiO_2/Carbon nanocomposite as a more effective electromagnetic

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wave absorbing material [7]. Therefore, this study was developed to enhance microwave absorption through $Ni_{0.5}Co_{0.5}TiO_3$ composites, with the addition of Polyvinyl Alcohol**.**

Microwaves (300 MHz–300 GHz) are used in various technologies, including volumetric heating-based drying [8]. Their interaction with materials depends on dielectric and magnetic properties, so optimal absorbing materials are needed [9]. The increase in electronic devices increases exposure to electromagnetic radiation which has an impact on the environment and health [10]. To overcome this, Radar Absorbing Materials (RAM) based on smart magnetic materials have been developed to absorb microwaves effectively, including in military applications. This composite utilizes metals such as nickel (Ni) and cobalt (Co), which have good conductivity and high corrosion resistance. However, Ni softens at high temperatures, while Co has low mechanical strength. To improve its mechanical properties and stability, polyvinyl alcohol (PVA) is used as a matrix due to its hydrophilic, flexible, and film-forming properties[11,12]. The synthesis process of this material involves mechanical milling, which allows for a more homogeneous particle distribution and improves its physical properties . With this approach, PVA-based $Ni_{0,5}Co_{0,5}TiO_3$ material is expected to be developed as a more efficient microwave absorber for high-tech applications.

X-ray diffraction spectroscopy (XRD) is a material characterization method for identifying crystalline phases, determining lattice structures, and measuring the particle size of nanocrystals. This technique studies the crystal structure, chemical composition, and physical properties of nanomaterials through the elastic scattering of X-ray photons by atoms in a periodic lattice [13]. A Scanning Electron Microscope (SEM) provides highly detailed images of sample surfaces through electron backscattering. SEM technology enables wide-area imaging, statistical analysis, and characterization of sample morphology and size distribution [14]. FTIR spectroscopy uses a Fourier transform to detect and analyze infrared spectra, providing molecular structure information through characteristic absorption bands. This method differentiates similar materials and is used to explore the reactions of extract compounds with HPMC-K4M polymer and chitosan [15]. Channel response describes the change in output to input in a communication channel, related to the scattering parameter S21=α, which indicates the ratio of output power to input. This study analyzes the magnitude characteristics of WBAN in a room with a uniform phase distribution. Measurements use a Vector Network Analyzer (VNA), where the initial magnitude in dB is linearized before being evaluated statistically. The measurement system includes a transmitting antenna (Tx) on the patient's body connected to port 1 of the VNA, and a receiving antenna (Rx) on the room divider connected to port 2 of the VNA to measure the S21 parameter [16]. From the measurements using the VNA, the RL value is calculated for each PVA concentration (10%, 20%, 30%) at a certain frequency.

2 Research and methodology

The research was conducted at the BJ. Habibie Science and Technology Area, Puspiptek, Serpong, South Tangerang, from May to December 2024. The materials used were $Ni_{0,5}Co_{0,5}TiO_3$, and PVA (purity ≥99%) with distilled water as solvent . Material synthesis was carried out by Mechanical Milling for 20 hours (ball:powder ratio 10:1), then sintered at 1000°C for 5 hours to produce $Ni_{0,5}Co_{0,5}TiO_3$. The composite was made by mixing

$Ni_{0,5}Co_{0,5}TiO_3$ in PVA solution (10 %,20%, 30%), then dried into tablet form. Characterization using XRD, SEM, FTIR, and VNA to analyze the structure, morphology, and microwave absorption. The results are presented in tables and graphs for evaluation.

3 Result and discussion

In this section, the research results are presented which include synthesis, characterization, and analysis of the ability of $Ni_{0,5}Co_{0,5}TiO_3$ material as a microwave absorbing composite. The discussion focuses on the crystal phase, morphology, microstructure, functional groups, and microwave absorption efficiency, by comparing the effect of variations in the concentration of active materials in the PVA matrix. The analysis is carried out to understand the relationship between structural parameters and material performance in applications as Radar Absorbing Material (RAM).

3.1 X-Ray diffraction (XRD)

The interaction between X-rays and the sample provides important data in XRD analysis. When X-rays hit electrons, as occurs in XRD imaging, the X-rays will be scattered. When X-rays hit the crystal lattice, X-ray diffraction occurs in various directions simultaneously because many atoms are exposed simultaneously [17]. The diffraction pattern of $Ni_{0,5}Co_{0,5}TiO_3$ powder in **Figure 1** shows a main peak at an angle of 2θ around 33°, corresponding to the (102) plane of the cubic spinel structure. This analysis is supported by other characteristic peaks, such as (102) at 23°, (110) at 35°, (113) at 40°, and several other peaks up to 84°. The increase in Ni^{2+} and Co^{2+} substitution causes a decrease in the intensity of the diffraction peaks and a shift in the 2θ angle according to Bragg's law, which indicates a change in the distance between crystals. The results of the XRD pattern refinement show that the $Ni_{0,5}Co_{0,5}TiO_3$ powder has a single phase without impurities, with a trigonal crystal system (rhombohedral axes) and space group R-3. The chi-square value of 1.582 indicates a good match between the data and the crystal model.

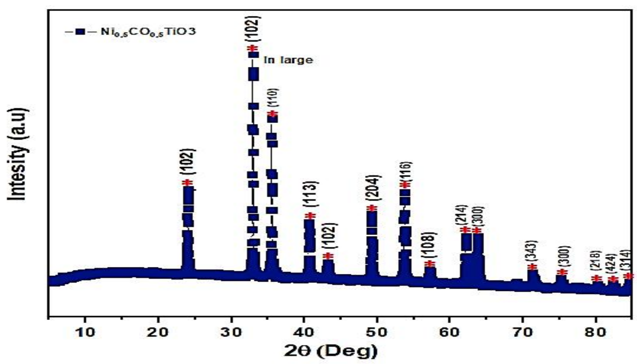


Fig. 1. XRD Diffraction Pattern of $Ni_{0,5}Co_{0,5}TiO_3$ Sample

In addition, XRD analysis will also be carried out on $Ni_{0,5}Co_{0,5}TiO_3$ composites with PVA polymers. The XRD method is used to identify the crystal structure through X-ray diffraction.

This tool can analyze the phase of powdered materials, solids, or amorphous and polycrystalline inorganic samples quantitatively and qualitatively, based on the 2θ angle, peak intensity, and lattice constant.

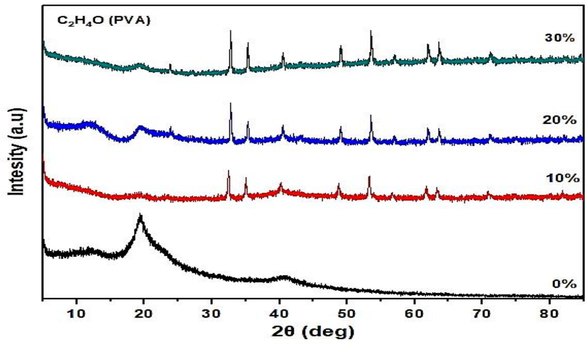


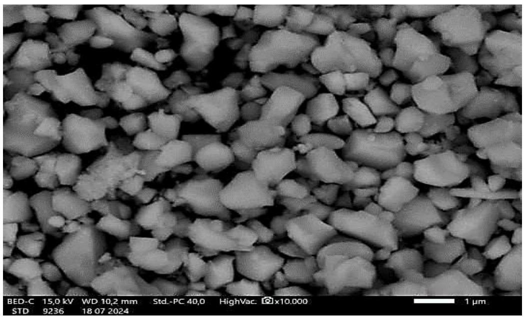
Fig. 2. XRD Diffraction Pattern of $Ni_{0.5}Co_{0.5}TiO_3$ Composite with PVA

The XRD diffraction pattern of $Ni_{0.5}Co_{0.5}TiO_3$ material with PVA in **Figure 2** shows sharp peaks that correspond to the structure of $Ni_{0.5}Co_{0.5}TiO_3$ based on the JCPDS database. The small shift in the peak position, as shown on the right side**,** reflects the change in lattice parameters due to the interaction between $Ni_{0.5}Co_{0.5}TiO_3$ and the PVA matrix, as well as the influence of mechanical strain during the High Energy Milling (HEM) process. The refinement results using the Rietveld method show nanometer grain size and low R-factor values, indicating a high agreement between the experimental data and the theoretical model. The diffraction pattern and fitting results confirm a homogeneous material with a single phase, which is important for microwave absorber applications.

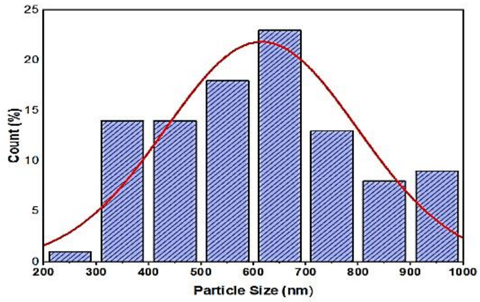
3.2 Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM) produces high-resolution images of a sample's surface by detecting electron backscatter showed in **Figure 3**. SEM testing allows for wide scanning and analysis of sample characteristics, including object counting and size distribution. Morphological images and concentrations of materials in a mixture can be obtained through SEM testing [18].

The surface morphology of $Ni_{0.5}Co_{0.5}TiO_3$ with clustered grains of various shapes and sizes. Nanometer-scale measurements indicate a homogeneous composition, aiding the analysis of surface roughness, grain size, and crystal structure. The particle size distribution was analyzed using ImageJ software by measuring 20-100 grains randomly to obtain an average nanometer size. High magnification reveals larger and uneven gaps, indicating a less homogeneous sample. The morphology also shows bright clumps of high atomic number elements and dark areas of low atomic number elements. The more homogeneous the sample, the higher its density.

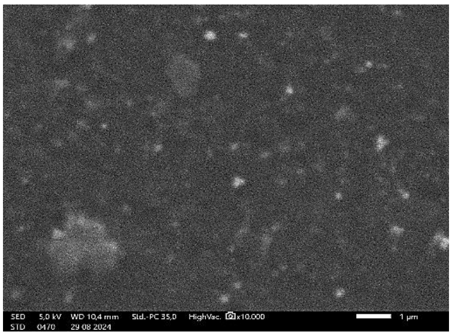


(a)



(b)

Fig. 3. (a) SEM Morphological Structure of $Ni_{0.5}Co_{0.5}TiO_3$ Material with 10000x Magnification, (b) Distribution of Particle Diameter Size of $Ni_{0.5}Co_{0.5}TiO_3$ Material



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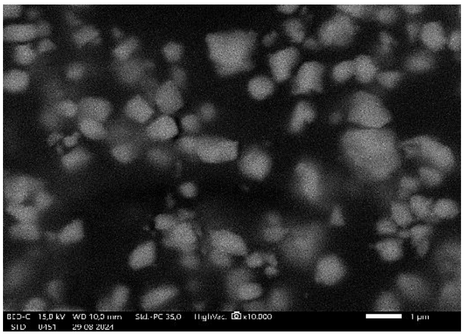

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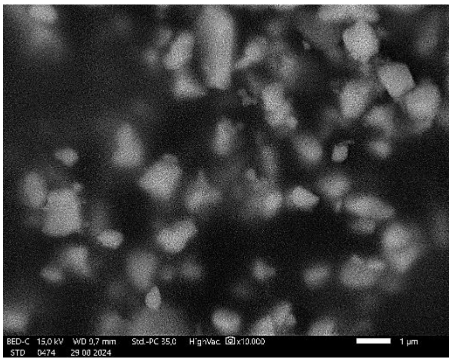

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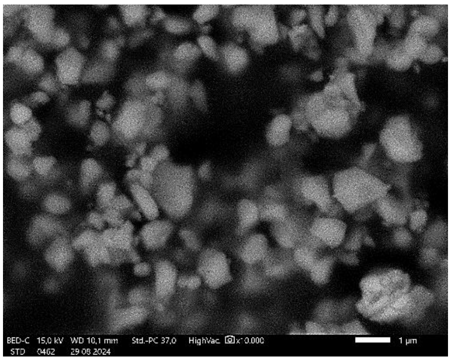
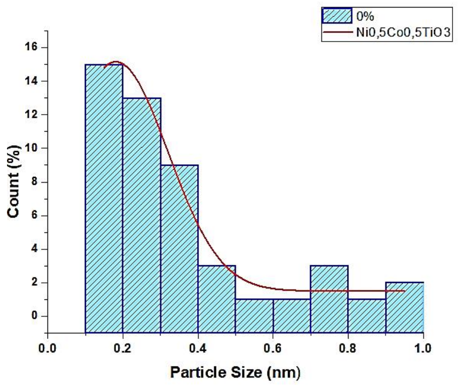


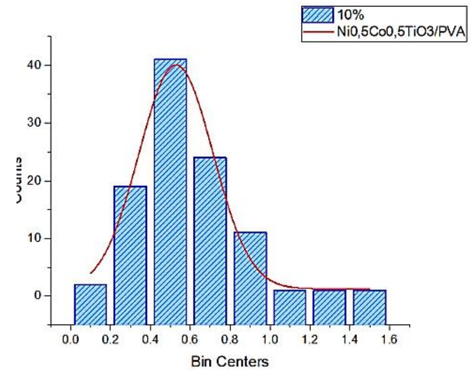
(d)

Fig. 4. SEM Morphological Structure of $Ni_{0.5}Co_{0.5}TiO_3$/PVA Material Magnification 10000x with Variations of (a) 0% (b) 10% (c) 20% (d) 30%

Characterization shows the morphological changes of $Ni_{0.5}Co_{0.5}TiO_3$ composites with various PVA levels. At 0 % PVA, particles tend to form large aggregates due to the lack of a binding matrix, resulting in a rough surface and uneven distribution. With the addition of 10 % PVA, particles are more distributed and aggregate size is reduced, improving the homogeneity of the composite. At 20% PVA, particles are more dispersed and the surface is smoother, reducing agglomeration and improving morphology. The addition of 30% PVA results in an optimal distribution with a thicker matrix, improving particle homogeneity and integration (**Figure 4**). Overall, PVA plays a role in reducing agglomeration and improving mechanical properties and electrical conductivity, supporting high-tech applications.



(a)



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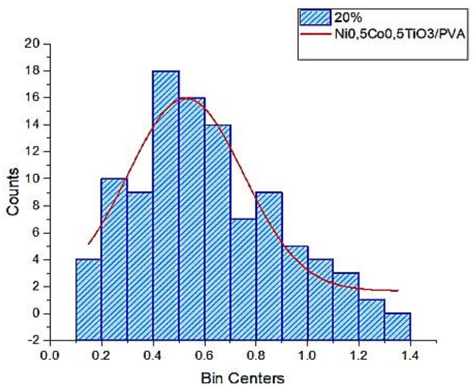

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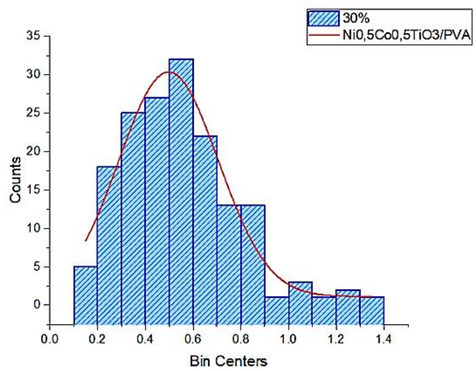


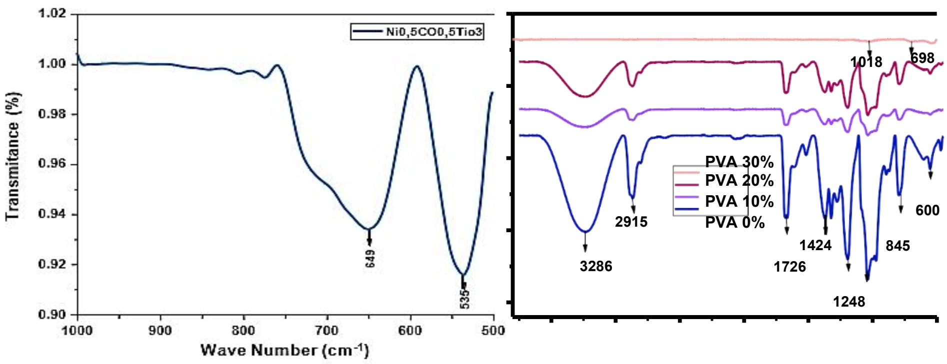
(d)

Fig. 5. Distribution of Particle Diameter Size of $Ni_{0.5}Co_{0.5}TiO_3$ Material with the addition of PVA with Variations of (a) 0%, (b) 10%, (c) 20% (d) 30%

The particle size distribution of $Ni_{0.5}Co_{0.5}TiO_3$ is affected by the addition of PVA. At 0% PVA, the particles form large aggregates with an uneven distribution. With 10% PVA, the particle size begins to become more uniform and aggregation decreases (**Figure 5**). At 20% PVA, the particles become more separated, with smaller diameters and a more homogeneous distribution. The addition of 30% PVA results in an optimal distribution, with the most uniform particle size and a smoother surface. Overall, increasing PVA improves homogeneity and reduces agglomeration, supporting the mechanical and functional properties of the material.

3.3 Fourier Transform Infrared (FTIR) characterization results

FTIR (Fourier Transform Infrared) is an infrared spectroscopy method that analyzes the interaction of infrared radiation with a sample to identify physical or chemical processes during mixing. This technique works in the range of 4000–400 $cm^{-}1$ and can be used quantitatively, because the absorbed energy is proportional to the concentration of the analyte. The fingerprint spectra indicate the wavenumber and chemical vibration bonds that absorb FTIR radiation [19].



(a) (b)

Fig. 6. (a) $Ni_{0.5}Co_{0.5}TiO_3$ FTIR sample, (b) $Ni_{0.5}Co_{0.5}TiO_3$ sample with the addition of PVA FTIR with variations of 0%, 10%, 20% and 30%

The FTIR spectrum results of $Ni_{0.5}Co_{0.5}TiO_3$ powder synthesized by mechanical milling using the KBr method were observed in the range of 500–1000 cm^{-1} . The spectrum in **Figure 6** showed two main peaks at 649 cm^{-1} and 535 cm^{-1}, which were related to the vibrations of metal ions in the composite spinel structure.

The 649 cm^{-1} peak is attributed to ion vibrations at octahedral positions, possibly originating from the O-Ti-O bond in TiO_6 or the interaction of Ti-O with Ni and Co. The 535 cm^{-1} peak indicates ion vibrations at tetrahedral positions, mainly involving Ni^{2+} and Co^{2+}. These absorption bands correspond to the infrared active modes of spinel materials, where v_1 (650–550 cm^{-1}) originates from tetrahedral metal-oxygen stretching vibrations, while v_2 (<550 cm^{-1}) from octahedral metal-oxygen vibrations [20].

The FTIR spectrum of $Ni_{0.5}Co_{0.5}TiO_3$ composite with varying PVA shows the interaction between $Ni_{0.5}Co_{0.5}TiO_3$ and PVA. At 0% PVA, the spectrum displays Ni-O, Co-O, and Ti-O vibrations, indicating the $Ni_{0.5}Co_{0.5}TiO_3$ phase. The addition of PVA (10%, 20%, 30%) gives rise to new absorption bands, such as OH (3200–3600 cm^{-1}), CH (2800–3000 cm^{-1}), and CO (1000–1300 cm^{-1}), with the intensity increasing with the PVA concentration. The shift or change in the intensity of the absorption bands of Ni-O, Co-O, and Ti-O indicates the possibility of chemical interaction between $Ni_{0.5}Co_{0.5}TiO_3$ and PVA. FTIR confirms the presence of both materials and indicates an interaction between them.

3.4 Vector Network Analyzer (VNA)

VNA measurements are performed to characterize the absorption properties of materials for microwaves. Reflection loss can be obtained by measuring diffusion parameters using a Vector Network Analyzer (VNA). Microwave absorption properties using VNA, the data provide results of the S_{11} value describing the value of energy reflected by the material [21].

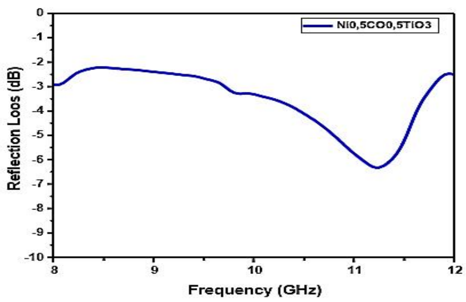


Fig. 7. Electromagnetic Wave Absorption Curve of $Ni_{0.5}Co_{0.5}TiO_3$ Powder

Microwave absorption of $Ni_{0.5}Co_{0.5}TiO_3$ powder synthesized by mechanical milling was measured by VNA in the range of 4.0–8.0 GHz in the reflection loss (RL) curve (**Figure 7**). The reflection loss indicates magnetic spin resonance which reflects the matching of impedance and microwave absorption (Sarwanto et al., 2019) [22]. In **Figure 7**, the $Ni_{0.5}Co_{0.5}TiO_3$ sample shows high absorption at 10.5–11.3 GHz with RL -2.92 dB to -6.33 dB, reaching 75.1% absorption at a frequency of 11.23 GHz.

Table 1. Results of reflection loss values of $Ni_{0.5}Co_{0.5}TiO_3$ powder samples synthesized using the mechanical milling method

Sample	Frequency (Ghz)	Reflection Loss (dB)	Though Power (%)
$Ni_{0.5}Co_{0.5}TiO_3$	11.23	-6.33	75.1%

Powder of $Ni_{0.5}Co_{0.5}TiO_3$ shows in **Table 1**, the phase formation greatly affects the magnitude of microwave absorption by the sample. The results of microwave absorption testing with VNA provide absorption of around 75.1% at a frequency of 11.23 GHz.

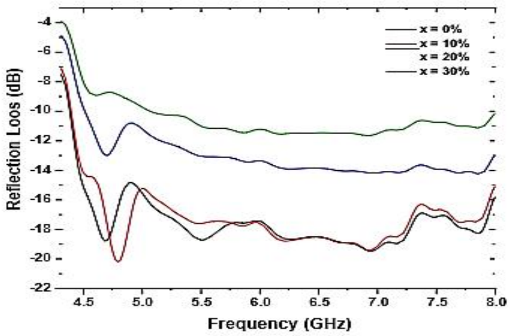


Fig. 8. Electromagnetic Wave Absorption Curve of $Ni_{0.5}Co_{0.5}TiO_3$ X-Band Composite with *Polyvinyl Alcohol* (PVA) (x = 0%, 10%, 20% and 30%)

Thus, the $Ni_{0.5}Co_{0.5}TiO_3$ sample can be applied quite well for several applications according to the needs that are carried out significantly as a material for microwave absorption. The reflection loss value will then be converted to percent absorption. **Figure 8** shows the composite of $Ni_{0.5}Co_{0.5}TiO_3$ with PVA has the highest absorption power of -20.1 dB at 4.79 GHz. The composition x = 10% is the most optimal in absorbing microwaves compared to others: x = 0% (-18.7 dB, 4.69 GHz), x = 20% (-13.0 dB, 4.70 GHz), and x = 30% (-8.98 dB, 4.61 GHz). The higher the PVA composition, the absorption power and frequency decrease. All samples have the potential as microwave absorbers according to their applications, depending on their respective maximum absorption frequencies.

Table 2. Results of *reflection loss values* of composite material samples $Ni_{0.5}Co_{0.5}TiO_3$ with Polyvinyl Alcohol (PVA) (x = 0%; 10%; 20%; 30%)

Sample	Frequency (Ghz)	Reflection Loss (dB)	Though Power (%)
0%	4.69	-18.7	99.92
10%	4.79	-20.1	99.99
20%	4.70	-13.0	96.51
30%	4.61	-8.98	87.96

Table 2 shows the reflection loss value of $Ni_{0.5}Co_{0.5}TiO_3$ composite at C-Band (4-8 GHz). Without $Ni_{0.5}Co_{0.5}TiO_3$ (x = 0%), the absorption power is -18.7 dB at 4.69 GHz. With the addition of $Ni_{0.5}Co_{0.5}TiO_3$, the highest absorption power occurs at x = 10% (-20.1 dB, 4.79 GHz), while x = 20% (-13.0 dB, 4.70 GHz) and x = 30% (-8.98 dB, 4.61 GHz) show a decrease. The more composition or thickness, the absorption power and frequency decrease. This material has potential for industrial applications, including as a strong film or composite material in commercial rubber clothing [23].

4 Conclusion

This study shows that $Ni_{0.5}Co_{0.5}TiO_3$ and PVA composite materials have great potential as microwave absorbers. XRD characterization confirms the perovskite structure, SEM shows homogeneous particle morphology, and FTIR confirms stable metal-oxygen bonds. RL results indicate that $Ni_{0.5}Co_{0.5}TiO_3$ powders have high absorption efficiency with RL values of around -6.33 dB, and the addition of PVA improves the absorption performance to RL -20.1 dB at a concentration of 10%.

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