

Synthesis and Characterization of ZnO@SiO₂ Composite for Microwave Absorber Applications

Muhammad Fauzan¹, Aditia Nur Bakti^{2**}, Elvina Trivida², Nova Nur Elisa Dewi¹, Djoko Triyono³, Iwan Sugihartono^{1*}

¹Physics Study Program, FMIPA, Universitas Negeri Jakarta, Jl. Rawamangun Muka, Jakarta, 13220, Indonesia

²Research Center for Testing Technology and Standards, National Research and Innovation Agency, Banten, 15314, Indonesia

³Physics Department, FMIPA, Universitas Indonesia, Kampus UI Depok, Depok, 16424, Indonesia

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This study aims to synthesize ZnO@SiO₂ composites via biosynthesis using *Moringa oleifera* leaf extract and to investigate the effect of different SiO₂ contents (pure ZnO, 1%, 3%, and 5% by weight) on their structural and microwave absorption properties, with ZnO prepared from a Zn(NO₃)₂·4H₂O precursor and calcined at 450 °C for 2 hours. The synthesized samples were characterized using X-ray Diffraction (XRD) and a Vector Network Analyzer (VNA). XRD results confirmed that all samples exhibited a hexagonal wurtzite crystal structure with space group P6₃mc and showed no secondary phases. The crystallite size decreased with increasing SiO₂ content, indicating nanoparticle formation and reduced crystallinity. VNA measurements showed that the ZnO@SiO₂ sample with 3% SiO₂ exhibited the best microwave absorption performance, with a minimum reflection loss (RL) value of -2.0251 dB at a frequency of 6.125 GHz. These results suggest that the 3% composition achieved better impedance matching and enhanced absorption efficiency.

Keywords: ZnO@SiO₂, biosynthesis, *moringa oleifera*, microwave absorption



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1. INTRODUCTION

The rapid development of advanced technology, particularly in defense and communication systems, has increased the demand for materials capable of mitigating electromagnetic interference (EMI) and reducing radar detection (Mathur et al., 2020). Radar Absorbing Materials (RAMs) play a crucial role in stealth technology by converting incident electromagnetic waves into thermal energy through dielectric and/or magnetic loss mechanisms, thereby minimizing reflected signals (Taryana et al., 2019). Among potential RAM candidates, zinc oxide (ZnO) has attracted significant interest due to its wide bandgap (~3.4 eV), high dielectric activity, non-toxicity, and environmental friendliness (Du et al., 2021). However, pure ZnO often suffers from particle agglomeration and limited structural stability, which can reduce its absorption efficiency. To address these limitations, silica (SiO₂) is introduced as a coating material to form a core-shell ZnO@SiO₂ structure. SiO₂, being an amorphous dielectric, can improve impedance matching, enhance particle dispersion, and induce interfacial polarization, thereby boosting microwave absorption performance (Yang et al., 2023). Conventional synthesis methods for ZnO@SiO₂ often involve hazardous chemicals and high energy consumption, raising environmental concerns. In contrast, green synthesis approaches utilizing plant extracts have emerged as sustainable alternatives, offering eco-friendly, low-cost, and biocompatible processes (Ahmed et al., 2017). *Moringa oleifera* leaves are particularly promising as a reducing and stabilizing agent due to their high content of bioactive compounds such as flavonoids, phenolics, and terpenoids, which can facilitate the reduction of metal ions and prevent nanoparticle aggregation (Sugihartono et al., 2024; Guda et al., 2023).

Contact Author: *Iwan-Sugihartono@unj.ac.id; ** adit006@brin.go.id

Previous studies have demonstrated that plant-mediated biosynthesis can produce nanoparticles with desirable structural and functional properties for various applications, including photocatalysis, sensing, and electromagnetic wave absorption (Asmathunisha & Kathiresan, 2012; Iravani, 2011). This study aims to synthesize ZnO@SiO₂ composites via biosynthesis using *Moringa oleifera* leaf extract and to investigate the effect of different SiO₂ contents (pure ZnO, 1%, 3%, and 5% by weight) on their structural, and microwave absorption properties. The synthesized materials were characterized using X-ray Diffraction (XRD) to determine crystal structure and crystallite size, and Vector Network Analyzer (VNA) for evaluating microwave absorption performance. The findings are expected to contribute to the development of environmentally friendly and cost-effective RAMs with optimized structural and electromagnetic properties for defense and EMI shielding applications.

2. METHOD

Moringa oleifera leaves were washed with distilled water, dried at room temperature for 7 days, ground into powder, and sieved. A total of 25 g of the powder was boiled in 250 mL of distilled water at 80 °C for 30 minutes, then filtered to obtain the extract. A zinc nitrate hexahydrate [Zn(NO₃)₂·4H₂O] solution with a concentration of 0.8 M was prepared and heated to 80 °C while being stirred. *Moringa oleifera* extract was added dropwise, and the pH was adjusted to 10 using NaOH. The mixture was stirred for 30 minutes to form a Zn(OH)₂ precipitate. The precipitate was washed with distilled water and ethanol, dried at 80 °C for 12 hours, and calcined at 450 °C for 2 hours to produce ZnO powder. The ZnO@SiO₂ composite was prepared by mixing 2 g of ZnO powder with SiO₂ powder at variations of (pure ZnO, 1%, 3%, and 5% by weight) in a mixture of 160 mL ethanol and 40 mL distilled water. The mixture was stirred for 30 minutes, and the pH was adjusted to 9–10 using NaOH. Stirring was continued for 4 hours at room temperature. The resulting precipitate was washed with distilled water and ethanol, dried at 80 °C for 12 hours, and ground into fine powder. X-ray Diffraction (XRD) was used to determine the crystal structure, and a Vector Network Analyzer (VNA) with the coaxial-line method in the frequency range of 4.3–8.5 GHz was used to measure microwave absorption properties.

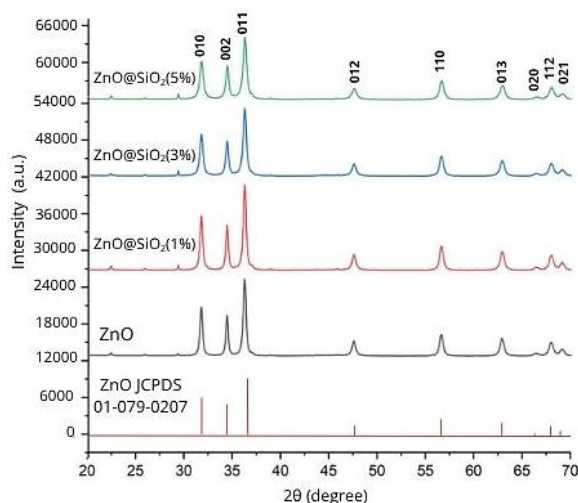


Figure 1. X-Ray Diffraction (XRD) pattern of ZnO@SiO₂ nanoparticle synthesis results with variations in SiO₂ concentration of (pure ZnO, 1%, 3%, and 5% by weight).

3. RESULTS AND DISCUSSION

3.1 Results

3.1.1 XRD

Figure 1 shows the X-ray diffraction (XRD) pattern of ZnO@SiO₂ material with variations in SiO₂ concentration of (pure ZnO, 1%, 3%, and 5% by weight). The diffraction pattern displays the highest intensity peaks on the (100), (002), and (101) planes, which are characteristic of the wurtzite-

type hexagonal crystal structure of ZnO. The XRD pattern does not show any additional peaks indicating a second phase or other impurity compounds. This indicates that the addition of SiO₂ does not change the main structure of ZnO and does not form new compounds. This finding is in line with the results of research by Godavarti et al. (2019), which stated that phase purity can be identified from the absence of foreign peaks in the diffraction pattern.

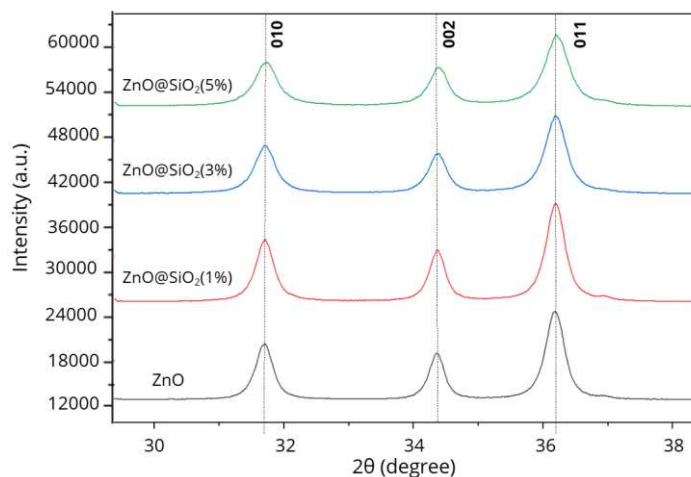


Figure 2. Diffraction angle shift of ZnO & ZnO@SiO₂ nanoparticles with variations in SiO₂ concentration of (1%, 3%, and 5%).

Based on Figure 2, the diffraction peaks for the (010), (002), and (011) planes of ZnO and the ZnO@SiO₂ composite (1%, 3%, and 5%) appear at similar 2θ angles, indicating that the addition of SiO₂ does not significantly change the d-spacing of the crystal planes. This indicates that the ZnO crystal structure remains stable after modification with SiO₂. XRD analysis was performed using HighScore Plus software, with reference data from JCPDS No. 01-079-0207, which refers to the hexagonal wurtzite ZnO structure. All samples exhibit diffraction patterns consistent with a hexagonal polycrystalline crystal structure, with the space group P6₃mc.

Table 1. Structural parameters of ZnO@SiO₂ nanoparticles with (SiO₂: pure ZnO, 1%, 3%, and 5% by weight).

Sampel	Structural Parameters				
	2θ (°)	Crystallite Size (nm)	d-spacing (Å)	FWHM (°)	Micro Strain (%)
hkl (010)					
ZnO	31.6631	37.25478	2.82357	0.3628	0.373641
ZnO@SiO ₂ (SiO ₂ : 1%)	31.672	31.76192	2.8228	0.3864	0.381694
ZnO@SiO ₂ (SiO ₂ : 3%)	31.6732	27.57151	2.82269	0.4392	0.441636
ZnO@SiO ₂ (SiO ₂ : 5%)	31.6886	16.02429	2.82136	0.5138	0.355121
hkl (002)					
ZnO	34.3246	33.08549	2.61048	0.3628	0.318229
ZnO@SiO ₂ (SiO ₂ : 1%)	34.3336	27.0398	2.60984	0.3864	0.30785
ZnO@SiO ₂ (SiO ₂ : 3%)	34.336	22.1056	2.60964	0.4392	0.336006
ZnO@SiO ₂ (SiO ₂ : 5%)	34.3526	14.95951	2.60842	0.5138	0.271795
hkl (011)					
ZnO	36.1505	50.40537	2.48271	0.3628	0.365447
ZnO@SiO ₂ (SiO ₂ : 1%)	36.1604	44.34186	2.48205	0.3864	0.384994
ZnO@SiO ₂ (SiO ₂ : 3%)	36.1662	39.66764	2.48159	0.4392	0.448642
ZnO@SiO ₂ (SiO ₂ : 5%)	36.1788	19.88283	2.48083	0.5138	0.40339

The decrease in crystallite size with increasing SiO₂ addition indicates that SiO₂ particles are able to inhibit the growth of ZnO grains, resulting in smaller and more finely distributed particles.

Table 2. Lattice parameters of ZnO@SiO₂ nanoparticles (SiO₂: pure ZnO, 1%, 3%, and 5% by weight).

Lattice Parameters	Sampel			
	ZnO	ZnO@SiO ₂ (SiO ₂ : 1%)	ZnO@SiO ₂ (SiO ₂ : 3%)	ZnO@SiO ₂ (SiO ₂ : 5%)
a = b (Å)	3.2496	3.2489	3.2488	3.2482
c (Å)	3.2496	5.204	5.2038	5.203
c/a	1.601791	1.601772	1.60176	1.60181
$\alpha = \beta$ (°)	90	90	90	90
γ (°)	120	120	120	120
Volume (Å ³)	47.6023	47.5708	47.5661	47.5412

3.1.2 VNA

Based on VNA measurements (Figure 3), the maximum and minimum RL values were obtained for the four samples, namely ZnO and ZnO@SiO₂ (SiO₂: 1%, 3%, and 5%), as shown in Table 3. The table shows that all samples exhibit microwave absorption capabilities, although the best absorption performance was obtained with the ZnO@SiO₂ (3%) composition. The ZnO@SiO₂ sample showed the largest minimum RL value, at -2.0251 dB at a frequency of 6.125 GHz, indicating the highest microwave absorption capacity. This indicates that the addition of 3% SiO₂ is the optimum composition in the ZnO@SiO₂ system for microwave absorption at that frequency. Conversely, a decrease in absorption performance occurred with the addition of 5% SiO₂, which could be caused by particle agglomeration or an imbalance in the material's microstructure.

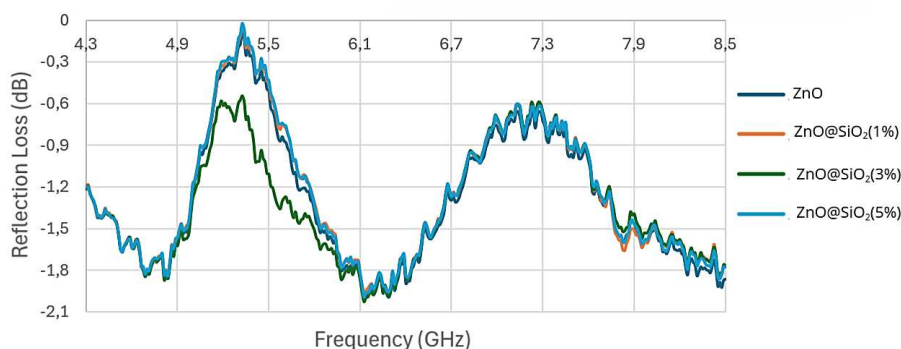


Figure 3. The relationship curve between the RL value and the frequency range of 4.3GHz - 8.5GHz for each sample, namely pure ZnO, and ZnO with the addition of SiO₂ of 1%, 3%, and 5%.

Table 3 Lattice parameters of ZnO & ZnO@SiO₂ nanoparticles (1%, 3% and 5%).

	Frequency (GHz)	ZnO	ZnO@SiO ₂ (SiO ₂ : 1%)	ZnO@SiO ₂ (SiO ₂ : 3%)	ZnO@SiO ₂ (SiO ₂ : 5%)
RL min (dB)	6.125	-1.9971	-1.9697	-2.251	-1.9934
RL max (dB)	5.325	-0.0842	-0.036	-0.5433	-0.0252

Figure 4 shows a comparison graph of the absorption levels of ZnO@SiO₂ (SiO₂: SiO₂: pure ZnO, 1%, 3%, and 5% by weight) and SiO₂/CNT (SiO₂: 0.5%, 1%, 2%), ZIF, 1% Ni/ZIF-67. To evaluate the microwave absorption performance of the ZnO@SiO₂ nanocomposite, a comparison was made with several comparative materials that had previously been studied at BRIN in 2024, namely SiO₂/CNT materials (with variations of 0.5%, 1%, and 2%), ZIF, and 1% Ni/ZIF-67.

The VNA results show that the ZnO@SiO₂ sample with a composition of 3% has the best microwave absorption performance, with a RL value of 2.0251 dB at a frequency of 6.125 GHz, demonstrating the best performance, among variations of ZnO & ZnO@SiO₂ (SiO₂: 1%, 5%), and other comparison materials such as SiO₂/CNT (SiO₂: pure ZnO, 1%, 3%, and 5% by weight), ZIF, 1% Ni/ZIF-67. This study shows that the best microwave absorption (RL) performance is in ZnO@SiO₂ with 3%

SiO₂. This indicates that the combination of this composite with a ZnO@SiO₂ core-shell structure is able to well combine the absorption strength of ZnO with the stability and smoothness of the morphology of SiO₂. This composite produces a good combination.

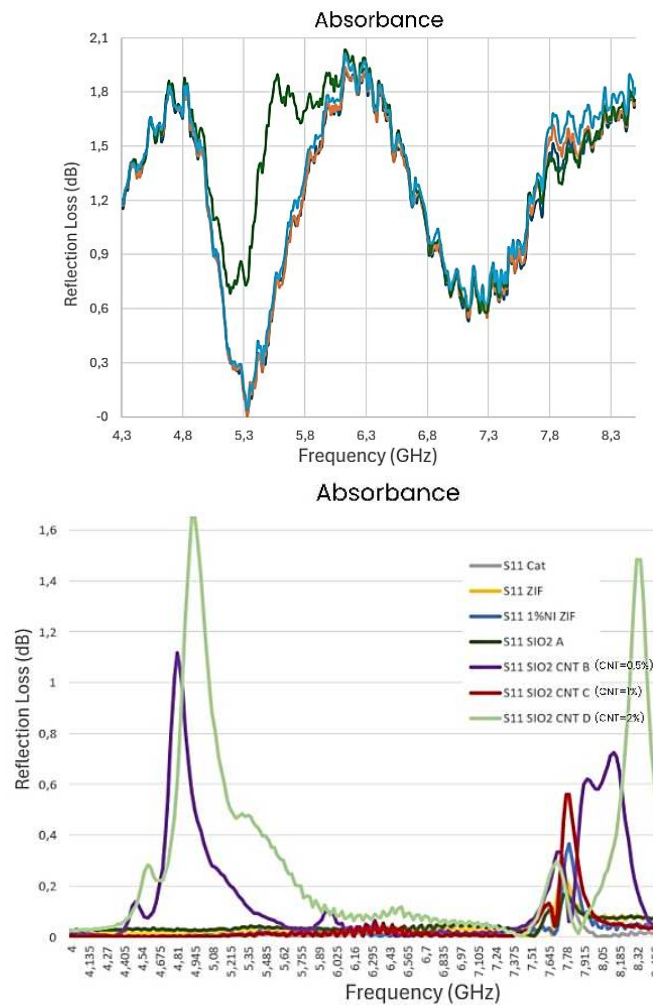


Figure 4. VNA Graph (S11 Parameter).

Table 4. Maximum and minimum RL values for ZnO & ZnO@SiO₂ (SiO₂: pure ZnO, 1%, 3%, and 5% by weight) S11 Parameter.

	Frequency (GHz)	ZnO	ZnO@SiO ₂ (SiO ₂ : 1%)	ZnO@SiO ₂ (SiO ₂ : 3%)	ZnO@SiO ₂ (SiO ₂ : 5%)
RL min (dB)	6.125	1.90484	1.935826179	2.03368	2.00917

Table 5. Maximum and minimum RL values for SiO₂/CNT.

	Frequency (GHz)	SiO ₂ /CNT (CNT: 2%)
RL min (dB)	4.9	1.675

3.2 Discussion

Based on the research data, the addition of varying SiO₂ concentrations in the ZnO@SiO₂ composite significantly influences the crystal structure, and microwave absorption capability of the resulting material. XRD characterization results indicate that all samples—both pure ZnO and

ZnO@SiO₂ with 1%, 3%, and 5% SiO₂—exhibit diffraction patterns characteristic of the hexagonal wurtzite crystal structure with a P6₃mc space group, without the presence of secondary phases. This confirms that the biosynthesis method using *Moringa oleifera* leaf extract did not alter the primary ZnO phase (Du et al., 2021). Crystallite size calculations using the Scherrer equation revealed a decreasing trend with increasing SiO₂ concentration. This reduction in crystallite size indicates finer particles, which can enhance the material's active surface area (Yang et al., 2023). This phenomenon is consistent with the role of SiO₂ as a shell in the core-shell structure, preventing excessive ZnO agglomeration and resulting in better particle dispersion. VNA measurements demonstrated that the sample with 3% SiO₂ achieved the best minimum Reflection Loss (RL) value of -2.0251 dB at a frequency of 6.125 GHz. This result indicates improved impedance matching and more effective interaction between the microwaves and the material surface. The presence of the SiO₂ layer is presumed to induce interfacial polarization, which enhances the dielectric loss mechanism and thereby strengthens microwave absorption. Thus, the XRD, SEM, and VNA data collectively support that varying SiO₂ concentrations affect both the structural and electromagnetic properties of ZnO@SiO₂ composites, with the 3% SiO₂ composition proving to be the optimum formulation in this study.

4. CONCLUSION

ZnO@SiO₂ composites synthesized via a biosynthesis method using *Moringa oleifera* leaf extract successfully retained the hexagonal wurtzite ZnO crystal structure without forming secondary phases, while the addition of SiO₂ reduced crystallite size, indicating the formation of nanoscale particles and preventing excessive agglomeration. Among the variations tested, the 3% SiO₂ sample exhibited the best microwave absorption performance with a minimum RL value of -2.0251 dB at 6.125 GHz, demonstrating improved impedance matching and higher absorption efficiency. These results indicate that a 3% SiO₂ composition optimally enhances both the structural and electromagnetic properties of ZnO@SiO₂, making it a promising, environmentally friendly, and cost-effective candidate for microwave absorber applications.

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