*In conjunction with the enhancement of societal living standards and the rapid development of information technology, an extensive variety of high-capacity electronic devices are being introduced to the market. The heightened demands result in the generation of electromagnetic wave radiation, which poses a potential risk to human well-being. Barium hexaferrite (BHF) is one of the radar-absorbing materials (RAMs) that can absorb electromagnetic waves because it has a high anisotropic field. However, its drawbacks are narrow absorption and less stability. Molybdenum disulfide (MoS2), is the best candidate for the reinforcement of BHF. The study investigated the impact of increasing the thiourea, temperature, hydrothermal holding time, and sample thickness on reflection loss. This study used a two-step molten salt and hydrothermal synthesis to make a BaFe* $_{12}O_{19}$ @MoS<sub>2</sub> *core-cell composite. Two-step molten salt and hydrothermal synthesis methods created single-phase BaFe12O19@MoS2 core-cell composites that worked well. The results showed that adding MoS2 to BHF changed BHF's magnetic properties from hard to soft. Increasing the hydrothermal temperature up to 220 °C effectively reduced the reflection loss of BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>. On a 2 mm thick sample containing 100 mmol thiourea, the study achieved an electromagnetic wave absorption of 99.97 % with a reflection loss of –35.41 dB (17.37 GHz). The results of this research can be applied to protect electronic devices vulnerable to signal interference from satellite radar systems at fre-*

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# **DEVELOPMENT OF BARIUM HEXAFERRITE CORE–SHELL COMPOSITES AS HIGH-PERFORMANCE MICROWAVE ABSORPTION BY OPTIMIZING HYDROTHERMAL SYNTHESIS**

# **Erlina Yustanti**

*Corresponding author*  Doctor of Materials Science, Professor\*, Head of the Laboratory Nanomaterial and Process Technology Laboratory\*\* E-mail: [erlina.yustanti@untirta.ac.id](mailto:erlina.yustanti@untirta.ac.id) **Alfian Noviyanto**

Doctor of Philosophy, Managing Director and Principal Investigator, Assistant Professor Center of Excellence Advanced Materials Nano Center Indonesia Raya Puspiptek str., A-12, Setu, Indonesia, 15314 Department of Mechanical Engineering Mercu Buana University Jend. Meruya Selatan str., Kebun Jeruk, Jakarta Indonesia, 11650 **Annisa Nur Fauziah** Bachelor of Engineering\* **Bachtiar Lubis** Bachelor of Engineering\* **Adhitya Trenggono** Master of Materials Science, Head of Department of Metallurgical Engineering\* Material Functional Laboratory\*\* **Ahmad Taufiq** Professor, Doctor of Physics, Head of Department of Physics Department of Physics Universitas Negeri Malang

Semarang str., 5, Malang, Indonesia, 65145 \*Department of Metallurgical Engineering\*\* \*\*Sultan Ageng Tirtayasa University Jenderal Sudirman str., 03, Cilegon, Banten, Indonesia, 42435

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*quencies of 12–18 GHz*

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#### **1. Introduction**

People are exposed to electromagnetic wave radiation from various sources, such as high-speed computers, cell phones, microwave ovens, radar, and satellite communication. The rapid development of wireless communication technology has been linked to a rise in public awareness regarding the dangers of electromagnetic pollution. The adverse effects of electromagnetic pollution on wireless devices, accurate instruments, and military safety make it imperative to promptly create high-performance radar-absorbing materials that absorb electromagnetic waves at wide frequencies. The study used key parameters to change the morphology of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite using hydrothermal synthesis to make it excellent at high-performance microwave absorption with the thinnest sample. However, the study focused on radar-absorbing materials at 12–18 GHz.

Barium hexaferrite (BHF) is a hard magnet with anisotropy, a high Curie temperature, a large magnetic field, good chemical stability, and low corrosion. Barium M-Hexaferrite (BaM), with its hexagonal crystal structure, is a permanent magnet with the potential to become a raw material for absorbing microwaves. These few decades have seen much research into M-type hexagonal ferrite because it has many uses. Barium hexaferrite is used in multiferroics, microwave absorbers, high-density magnetic recording media, telecommunications equipment, and magneto-optics[1]. Ferrite nanoparticles are essential in science and technology because they are adsorptive, magnetic, and catalytic. Nanoparticles have large surface areas, ferrite-based materials have good thermal stability and mechanical properties, and their chemical composition is easy to engineer. It can be controlled to improve their performance [2]. BaM is a ferromagnetic oxide with dielectric and magnetic properties often applied to RF (radio frequency) and microwaves. The use of BaM as a permanent magnet and magnetic recorder is in great demand, so research about this continues to be improved.

The researchers previously developed anti-radar material with a more than 2.5 mm thickness. The results were achieved through a complicated and inefficient preparation process, combining various synthesis methods and layered composite techniques to form a core@shel@shell structure. Therefore, the studies are devoted to creating the thinnest sample of radar absorber material with maximum electromagnetic wave absorption with efficient preparations are scientific relevance.

#### **2. Literature review and problem statement**

In a previous study conducted by [3], sol-gel auto-combustion confirmed the successful substitution of Er–Cr cations in the  $SrEr_xCr_xFe_{12-2x}O_{19}$  achieved maximum reflection loss of −34.71 dB at *x*=0.5. However, this study does not explain the influence of the  $\rm{SrEr_xCr_xFe_{12\text{-}2x}O_{19}}$  morphological structure on the increased absorption of electromagnetic waves. Radar-absorbing materials can be created by producing barium hexaferrite from the top down or bottom up. High-power ultrasonic irradiation and mechanical alloying created  $BaFe<sub>9</sub>Mn<sub>1.5</sub>Ti<sub>1.5</sub>O<sub>19</sub>$ with 90 % electromagnetic absorption and a reflection loss of –19.75 dB at 13.6 GHz [4]. Solid-state methods offer the benefits of waste-free production. However, important investigated morphological structure for over 90 % of electromagnetic wave absorption, which is unresolved in this paper. In addition, the paper [5] conducted an approach through a solid reaction that resulted in a reflection loss of −32.1 dB at 11.2 GHz. The study highlights the use of  $Co^{3+}$  at  $x=0.4$  successfully synthesized using the thinnest sample,  $BaFe_{12-x}Co_xO_{19}$ . This research is superior in the x-band, but electromagnetic wave absorption is not optimal in the Ku band. Chemical vapour deposition with pyrolysis temperature control demonstrated that Fe@Fe3C@CNTs ternary nanohybrids have significantly improved electromagnetic wave absorption capabilities [6]. The morphological samples were analyzed with variations in the pyrolysis temperature, while the hydrothermal process was conducted at 200 °C for 12 hours. This study did not examine the morphological impacts on temperature and hydrothermal holding time.

Previous researchers [7] successfully employed a hydrothermal synthesis technique to reduce reflection loss signifi-

cantly to a remarkable value of −61 dB. This achievement was observed at a sample thickness of 1.7 mm. However, the influence of temperature and hydrothermal holding time has yet to be investigated. The production of  $\mathrm{BaFe_{11.6}Mg_{0.2}Al_{0.2}O_{19}}$  by a co-precipitation technique, employing a controlled calcination temperature of 900 °C, resulted in a reflection loss of −58.60 dB at a frequency of 10.91 GHz [8]. Due to the common usage of radar-absorbent material as a coating, this study did not conduct additional examination on differences in sample thickness. Therefore, it focused on samples with a thin thickness.

Several research studies have highlighted the significance of the mass ratio composition in determining the success of enhanced electromagnetic wave absorption. The practical synthesis of a multi-component composite using solvothermal fabrication was reported. The composite consisted of a core material, Fe<sub>3</sub>O<sub>4</sub>@C, and a shell material, MoS<sub>2</sub>. It was shown that the microwave absorption behaviour of the composite could be easily modified by altering the mass ratio of the components [9]. The described multi-component composite technique demonstrates that raising the sample thickness from 2 to 3 mm increases reflection loss. These observations are interesting for further analysis since increased sample thickness generally leads to decreased reflection loss. As a result, developing a more uncomplicated encapsulating technique with comparable outcomes is interesting. Another researcher also reported based on electromagnetic characteristics, it was discovered that the Fe@  $Fe<sub>3</sub>O<sub>4</sub>$  to MoS<sub>2</sub> mass ratio had a significant influence on the absorption and bandwidth [10]. Due to the increasing sample thickness, the reflection loss has shifted from the Ku band to the C band. Furthermore, additional improvements are needed to stabilize the increased sample thickness on the Ku Band by controlling the sample's morphology.

The researchers later developed a lot of layered composite techniques. Using double-layered absorbing structures proved to be a highly efficient method for enlarging absorption bandwidth. A sophisticated process that included hummers for RGO creation, hydrothermal fabrication, and hybrid structure production through co-precipitation produced  $MoS_2/RGO$  hybrids. Although  $MoS_2/RGO$  composites with increased microwave absorption properties have been reported [11]. However, this method use is limited due to the complicated and inefficient preparation process.

All this suggests that it is advisable to conduct a study investigating the influence of temperature and hydrothermal holding time, which researchers have yet to explore. The  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  morphology also found an unresolved problem: the emergence of nanorod structures that block increased absorption of electromagnetic waves. Therefore, it is crucial to control the influence of several factors, such as hydrothermal temperature, holding time, thiourea composition, and sample thickness, to prevent the emergence of nanorods that inhibit the absorption of electromagnetic waves and enhance the absorption in the thinnest sample. This study developed a core-shell structure using a combination of molten salt and hydrothermal synthesis. This method is widely used in several industries due to its practicality, accessibility, cost-effectiveness, and efficiency compared to other methods.

#### **3. The aim and objectives of the study**

The study aims to develop techniques to improve the absorption of electromagnetic waves in barium hexaferrite with the thinnest sample. This will make it possible to apply it as a coating material for electronic devices that require a thin anti-radar layer.

To achieve this aim, the following objectives are accomplished:

– to investigate the crystal structure of the  $BaFe_{12}O_{19}$ @  $MoS<sub>2</sub> core-shell composite;$ 

– to investigate the morphology of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite;

– to evaluate the magnetic properties of the  $BaFe_{12}O_{19}$ @ MoS2 core-shell composite;

– to investigate anti-radar capability.

#### **4. Materials and methods**

#### **4. 1. Object and hypothesis of the study**

The object of this study is to develop a technique for generating anti-radar material by using a coating material with the minimum possible sample thickness. The hypothesis proposes that by creating a core-shell composite, the crystal structure can be modified to enhance its ability to absorb electromagnetic waves. These modifications will reduce the adverse effects

of electromagnetic waves on anti-radar material by minimizing their transmission and reflection. The premise underlying this work is that using  $MoS<sub>2</sub>$  as a dielectric material results in a significant decrease in reflection loss, hence optimizing the absorption of electromagnetic waves. The hydrothermal treatment's temperature and holding time significantly affect how well the core-shell composite is formed, especially when making the petals the right thickness and depth. This configuration facilitates a more concise coreshell synthesis, resulting in nearly 100 % absorption of electromagnetic waves.

The composition ratio of  $MoS_2:BaFe_{12}O_{19}$ was changed in this study, along with the sample thickness, temperature, and holding time, during the hydrothermal synthesis process. The efficiency of reduced reflection loss in comparison to some of the synthesis factors was analyzed.

#### **4. 2. Materials**

Molten salt synthesis used a mixture of precursors: barium chloride dihydrate  $(BaCl<sub>2</sub>.2H<sub>2</sub>O, \geq 99 %$ , Merck), iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>,  $\geq$ 99%, Sigma-Aldrich), barium carbonate (BaCO<sub>3</sub>,  $\geq$ 99 %, Pudak Scientific), and sodium chloride (NaCl, ≥99 %, Pudak Scientific). Hydrothermal synthesis used  $BaFe<sub>12</sub>O<sub>19</sub>$ resulting from molten salt synthesis with thiourea (CH<sub>4</sub>N<sub>2</sub>S,  $\geq$ 99%, Loba Chemie), ammonium heptamolybdate tetrahydrate  $((NH_4)_6Mo_7O_{24}.4H_2O, \geq99\%$ , Pudak Scientific), and deionized water from water-one.

# **4. 3. Synthesis of BaFe12O19@ MoS2 core-shell composite**

In this study, two steps of molten salt synthesis and then hydrothermal synthesis were responsible for controlling the structure of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core-cell composite. The first step in making molten salt involved mixing  $BaCO<sub>3</sub>$ , Fe<sub>2</sub>O<sub>3</sub>, and NaCl in a 1:2 mass ratio for  $BaFe_{12}O_{19}$ :NaCl. The precursors were milled using an alcohol medium at a speed of 150 rpm for 2 h, and then the wet milling product was calcined at 1000 °C for 2 h. In the second molten salt, there was a mixture of precursors of  $Fe<sub>2</sub>O<sub>3</sub>$  and  $BaCl<sub>2</sub>.2H<sub>2</sub>O$  with a mass ratio of 1:2 and  $BaFe<sub>12</sub>O<sub>19</sub>:Fe<sub>2</sub>O<sub>3</sub>$  with a molar ratio of 1:500. The precursor mixture was milled at 150 rpm for 2 h. The sample was calcined at a temperature of 1100 °C for 8 h. In the synthesis of the  $BaFe_{12}O_{19}QMoS_2$  core-shell composite, there was a mixture of 1 mmol  $(NH_4)_6M_0T_2A_4H_2O$ , thiourea with a variation of (10, 20, 50, and 100) mmol, 35 ml deionized water, and 0.2 gr BHF. Then, the mixture was brought to the hydrothermal process at various temperatures (160, 180, 200, and 220) °C and holding times (14, 16, 18, and 20) h. A schematic illustration of the molten salt and hydrothermal synthesis of  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$ core-cell composite is shown in Fig. 1.

Table 1 lists all the codes used to identify the samples from the hydrothermal synthesis processes.



Fig. 1. Schematic of  $BaFe_{12}O_{19}$  core-shell composite fabrication

Table 1

Code identification and treatment for all samples

Sample code	Thiourea composition at	Sample thick-	Hydrothermal	Hydrothermal
	$BaFe12O19@MoS2$ (mmol)	ness(mm)	temperature $(^{\circ}C)$	holding time (h)
BaFe <sub>12</sub> O <sub>19</sub>		2		
MoS <sub>2</sub>	30	2	220	18
$C_1$	10	$\overline{2}$	220	18
C <sub>2</sub>	20	$\overline{2}$	220	18
$C_5$	50	2	220	18
$C_{10}/T_{22}/H_{18}$	100	1.0; 1.5; 2.0; 2.5	220	18
$T_{16}$	100	2	160	18
$T_{18}$	100	$\overline{2}$	180	18
$T_{20}$	100	$\overline{2}$	200	18
$H_{14}$	100	$\overline{2}$	220	14
$H_{16}$	100	$\overline{2}$	220	16
$H_{20}$	100	2	220	20

C is the sample code  $BaFe_{12}O_{19}QMoS_2$  with a composition variation of thiourea.

T is the sample code  $BaFe_{12}O_{19}@MoS_2$  with hydrothermal temperature variations.

H is the sample code  $BaFe_{12}O_{19}@MoS_2$  with hydrothermal holding time variation.

As for variation in sample thickness in representative sample code  $C_{10}/H_{22}/T_{18}$ .

## 4.4. Characterization of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core**shell composite**

The crystal structure was evaluated using x-ray diffraction (XRD, Bruker D8 Advance ECO with Lynxeye XE-T Cu Source) and scanning electron microscopy-energy dispersive x-ray spectroscopy (SEM-EDS, Carl Zeiss EVO 10) to determine the composition and morphology of the samples. Particle size distribution was analyzed using a particle size analyzer (PSA, Malvern Zetasizer Pro Blue Dynamic Light Scattering Instrument). Hysteresis loop analysis used a vibrating sample magnetometer (VSM, 1.2H, Oxford). The radar-absorbing materials (RAMs) performance was analyzed using a vector network analyzer (VNA, two ports, Anritsu MS 46322 A, Allen, TX, USA). The absorption power of EM waves was calculated based on reflection loss through the data generated by the transmission signal (S21) and the reflection signal (S11). The reflection loss (RL) was calculated using the equations of Nicholson Ross Weir [12–19] shown in equations (1) and (2):

$$
Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\bigg[ j\bigg(\frac{2\pi fd}{c}\bigg) \sqrt{\mu_r \varepsilon_r} \bigg],\tag{1}
$$

$$
RL(\text{dB}) = 20 \log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|.
$$
 (2)

The values ε and µ represent the relative permittivity and permeability, velocity of light in vacuum (*c*), electromagnetic

frequency (*f*), sample thickness (*d*), impedance material  $(Z_{in})$ , and free space impedance  $(Z_0)$ .

#### **5. Experimental results of synthesizing BaFe12O19@MoS2 core-shell composite**

## 5. **1.** Crystal structure of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core**shell composite**

Fig. 2 compares the diffraction patterns of  $BaFe<sub>12</sub>O<sub>19</sub>$ ,  $MoS<sub>2</sub>$ , and  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  core-shell composite.

Fig. 2 shows how the diffraction patterns of  $BaFe_{12}O_{19}$ and  $MoS<sub>2</sub>$  in the standard database matched those of samples made of  $BaFe_{12}O_{19}$ ,  $MoS_2$ , and a core-shell composite of BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>. Fig. 2 shows that BaFe<sub>12</sub>O<sub>19</sub>, MoS<sub>2</sub>, and the  $BaFe_{12}O_{19}@MoS_2$  core-shell composite have a single phase. The Crystallography Open Database (COD) number for  $BaFe_{12}O_{19}$  is 1008841, and the COD number for  $MoS<sub>2</sub>$  is 1010993. Table 2 shows the results of the Rietveld refinement of BaFe<sub>12</sub>O<sub>19</sub>, MoS<sub>2</sub>, BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> (C<sub>10</sub>), which has a hexagonal crystal structure.  $BaFe_{12}O_{19}@MoS_2$ has space group P63/mmc, *a*=5.8924 Å, and *b*=23.1989 Å, which are the same as the results of a previous study [20].

Table 2

Rietveld refinement of  $BaFe_{12}O_{19}$ , MoS<sub>2</sub>, and  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite

Sample	BaFe <sub>12</sub> O <sub>19</sub>	MoS <sub>2</sub>	$BaFe_{12}O_{19}QMoS_2(C_{10})$
<b>SIG</b>	0.2315	0.62803	1.2952
$Rwp(\%)$	17.9527	2.2720	6.6653
$\text{Rexp}$ (%)	13.1957	12.2124	5.1761
$a(\AA)$	5.8948	3.1439	5.8924
c(A)	23.2129	12.3151	23.1989
Volume of unit cell $(\AA^3)$	697.1736	107.708	697.5686
Density $(g/cm^3)$	5.2841	4.8800	5.2900



Fig. 2. The diffraction patterns of  $BaFe_{12}O_{19}$ , MoS<sub>2</sub>, and  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite

Table 2 shows the density of  $BaFe_{12}O_{19}$  of 5.29 g/cm<sup>3</sup> is in line with the results of a previous study [21]. Furthermore, MoS2 (space group=P63/mmc, *a*=3.1439 Å, *c*=12.3151 Å) is consistent with a previous study [22].

## 5.2. The morphology of the  $BaFe_{12}O_{19}@MoS_2$  core**shell composite**

The surface morphology of the samples and the elemental composition of these materials were identified using SEM-EDS. Fig. 3 shows the EDS mapping to determine the composition and distribution of elements in the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite (C<sub>10</sub>) has a homogeneous distribution.



Fig. 3. The energy dispersive x-ray spectroscopy mapping of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> (C<sub>10</sub>) core-shell composite

As seen in Fig. 4 Spectrum 1, the detailed intensity of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>(C<sub>10</sub>)$  elemental composition.

Including the Au element within the  $BaFe_{12}O_{19}@MoS_2$  $(C_{10})$  structure is feasible due to using Au as a coating during the sample preparation process. In order to achieve optimal imaging for non-conductive materials, it is typically necessary to use gold and carbon as coating elements. Table 3 summarizes the composition expressed in weight percentage (wt. %) and atomic percentage (at %) as depicted in Spectrum 1 of Fig. 4.

Fig. 5 shows the results of SEM characterization at 30,000 and 60,000 times magnification. Based on Fig. 5, *a* the structure morphology of the single-phase  $BaFe_{12}O_{19}$  is a hexagonal plate-like crystal structure. Then, Fig. 5, *b* shows the single-phase nanoflower  $MoS_2$ , while Fig. 5,  $c-f$  shows the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> composites (C<sub>1</sub>–C<sub>10</sub>).

The results of the particle size distribution analysis performed with ImageJ software are summarized in Fig. 6. An increase in the thiourea composition of the  $BaFe_{12}O_{19}@MoS_2$ core-shell composite increases the particle size. Based on the surface morphology of  $BaFe_{12}O_{19}@MoS_2$ , an increase in thiourea composition causes more  $MoS<sub>2</sub>$  to cover the surface of BHF. It was confirmed that the thiourea content was higher,

> which led to more agglomeration and  $MoS<sub>2</sub>$ growth on the surface of  $BaFe_{12}O_{19}$ . According to a previous study [7], when the amount of thiourea goes up, the growth pathway for MoS2 nanoflowers on the BHF surface also gets longer and denser. According to Fig. 5, *f*,  $BasFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> (C<sub>10</sub>) has a distinctive$ morphology different from the other products containing the other thiourea compositions. The geometry of the nanoflower, which was suggested and made, could make it possible to get both a large contact area for intense interfacial polarization and a more significant number of places where electromagnetic waves could reflect and scatter[16]. An increasing concentration of thiourea increases the thickness of  $MoS<sub>2</sub>$ , enhancing its ability to absorb electromagnetic waves on the BHF surface. Fig. 5, *f* demonstrates the maximum identified thickness and density of  $MoS<sub>2</sub>$  for the  $C<sub>10</sub>$ .

> > Table 3

# The element mapping of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite  $(C_{10})$





Fig. 4. The elemental content of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core-shell composite (C<sub>10</sub>)



Fig. 5. The micrograph of particles size:  $a - BaFe_{12}O_{19}$ ;  $b - MoS_2$ ;  $c - f - BaFe_{12}O_{19}$ @MoS<sub>2</sub> (C<sub>1</sub>–C<sub>10</sub>)



Fig. 6. The particle size of  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite by using ImageJ analysis

The findings of SEM analysis of  $BaFe_{12}O_{19}@MoS_2 (C_{10})$ to study the effect of temperature and hydrothermal holding time variations on the production of  $MoS<sub>2</sub>$  nanoflowers on the BHF surface are shown in Fig. 7. The particle size of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  core-shell composite grows as the hydrothermal temperature rises (Fig. 7, *a–d*). As the hydrothermal holding time goes up, on the other hand, the nanoflower pathways on the surface of BHF tend to get bigger. The  $MoS<sub>2</sub>$ nanoflower petals also get denser, thicker, and more profound.

Fig. 7 illustrates the impact of elevated hydrothermal temperature on the morphology of a core-shell composite consisting of  $BaFe_{12}O_{19}$ @MoS<sub>2</sub>. The process of MoS<sub>2</sub> nanorod creation initiates at a temperature of 160 °C, as depicted in Fig. 7, *b*. Subsequently, the temperature is raised to 180 °C and ultimately reaches its maximum at 200 °C. At a hydrothermal temperature of 220°C, a hexagonal  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  floral structure may be observed (Fig. 7, *a*). The presence of the nanorod structure during synthesis is considered disadvantageous due to its disruptive effect on the electromagnetic wave absorption process. The presented figure illustrates the morphology of the sample free of nanorods when subjected to a hydrother-

mal temperature of 220 °C. Based on a previous study [13], nanowires were formed from nanorods at a temperature of 200 °C using hydrothermal synthesis. Decreasing the hydrothermal temperature increases the unwanted growth of MoS2 nanorods. BaFe12O19@MoS2 core-shell composite synthesis at a low temperature of 180 °C produces  $MoS<sub>2</sub>$ with nanorods and nanotube structures [23].

An analysis of particle size distribution was conducted using PSA. Brownian motion is the fundamental concept of PSA; when a group of particles scatters light, the scattering angle will be inversely proportional to the size of the particles. The crystallites' size and the sample's morphological structure depend on the precursor's composition, the solution's pH, and the temperature [24]. The particle size of the  $BaFe<sub>12</sub>O<sub>19</sub>@$  $MoS<sub>2</sub> core-shell composite increases with increasing thiourea$ composition, as seen in Fig. 8. The ImageJ analysis results of SEM and PSA characterizations are reported in Table 4.

The particle size determined using PSA characterization, as shown in Table 4, follows the same trends and is not significantly different from the particle size determined using SEM, as shown in Fig. 6. Table 4 shows the PSA measurement findings, which demonstrate a polydispersity index (PI) in the range of 0.3–0.7. It demonstrates that the BaFe12O19@MoS2 core-shell composite exhibits non-uniform particle sizes, various shapes, and a broad particle distribution; samples  $C_1$  and  $C_5$  even have bimodal curves.

Table 4

Particle size of  $BaFe_{12}O_{19}$ ,  $MoS_2$  and  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> coreshell composite based on SEM and PSA analysis

Sample	$SEM$ (nm)	PSA(nm)	Polydisperse index	
BaFe <sub>12</sub> O <sub>19</sub>	485.6	410	0.4	
MoS <sub>2</sub>	345.6	287	0.4	
C <sub>1</sub>	309.50	356	0.6	
$C_{2}$	426.41	415	0.3	
$C_5$	686.41	660	0.7	
$\mathbb{C}_{10}$	723.25	847	0.7	



Fig. 7. The morphology of BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>:  $a$  – samples of C<sub>10</sub>/T<sub>22</sub>/H<sub>18</sub>;  $b-d$  – hydrothermal temperatures of T<sub>16</sub>, T<sub>18</sub>, and T<sub>20</sub>;  $e-g$  – hydrothermal holding times of H<sub>14</sub>, H<sub>16</sub>, and H<sub>20</sub> h – histogram of particle size distribution



Fig. 8. Particle size distribution of the  $BaFe_{12}O_{19}$ ,  $MoS_{2}$ ,  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$ 

Fig. 9 shows that the increase in hydrothermal temperature is directly related to the increase in particle size and the widening of the particle size distribution of the BaFe12O19@MoS2 core-shell composite. An increase in hydrothermal temperature from 160°C to 220 °C increases the *z*-average particle size from 257.5 nm to 847 nm.

Particle size growth is affected by the nucleation of the  $BaFe_{12}O_{19}@MoS_2$  core-shell composite, which becomes more numerous when the hydrothermal temperature increases; this is also confirmed by a previous study [25, 26].

The influence of different hydrothermal holding durations on the particle size distribution of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$ core-shell composite is shown in Fig. 10.

The increase in the nucleation rate of the  $BaFe_{12}O_{19}$  @MoS<sub>2</sub> core-shell composite and the increase in hydrothermal holding time can increase the z-average from 174.2 nm to 993 nm. The increase in hydrothermal holding time is directly proportional to the increase in the size of the  $MoS<sub>2</sub>$  nanoflower; this is following a previous study [26].



Fig. 9. Particle size distribution of  $BaFe_{12}O_{19}@MoS_2$  coreshell composite at various hydrothermal temperatures





Table 5 presents the particle sizes of  $BaFe_{12}O_{19}$ ,  $MoS_2$ , and  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  core-shell composites under different hydrothermal temperatures and holding times. This table compares the particle sizes obtained using scanning electron microscopy (SEM) and particle size analyzer (PSA) techniques.

Particle sizes of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composites at various hydrothermal temperatures and holding times

Table 5



Table 5 compares the particle sizes obtained using scanning electron microscopy (SEM) and particle size analysis (PSA) techniques.

# **5. 3.** The magnetic properties of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> **core-shell composite**

The objective of employing a vibrating sample magnetometer is to analyze and determine the magnetic characteristics of BaFe<sub>12</sub>O<sub>19</sub> and BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>, as illustrated in Fig. 11.



Fig. 11. Loop hysteresis of the BaFe<sub>12</sub>O<sub>19</sub> and BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core-shell composites at various thiourea compositions

Fig. 11 shows that  $BaFe<sub>12</sub>O<sub>19</sub>$  has the greatest magnetic saturation and remanence. Increasing thiourea composition  $(C_1-C_{10})$  significantly decreases magnetic saturation and remanence. A decrease in magnetic saturation, remanence, and coercivity shows that  $BaFe_{12}O_{19}@MoS_{2}$ 's properties change from those of a hard magnet to those of a soft magnet.

## **5. 4. Analysis of radar absorber performance using a vector network analyzer**

A vector network analyzer (VNA) is used to prove the absorption performance of RAMs. VNA characterization results in the permeability and permittivity of real and imaginary. Real permeability and real permittivity represent the storage of electric and magnetic energy, while imaginary permeability and imaginary permittivity represent dielectric losses and magnetic losses in the sample [27]. Radar-absorb-

ing material performance is calculated using reflection loss equations (1) and (2). Fig. 12 shows the reflection loss (RL) of BaFe<sub>12</sub>O<sub>19</sub>, MoS<sub>2</sub>, and BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> (C<sub>1</sub>-C<sub>10</sub>) at various thiourea compositions at a sample thickness of 2 mm.



Fig. 12. Reflection loss of the BaFe<sub>12</sub>O<sub>19</sub>, MoS<sub>2</sub>, BaFe<sub>12</sub>O<sub>19</sub>@ MoS2 core-shell composites at various thiourea compositions

Increasing thiourea composition reduces the RL of  $BaFe_{12}O_{19}$  $@MoS_2$  as much as 149.01%, which is from  $-14.22$  dB (13.65 GHz) to  $-35.41$  dB (17.37 GHz). The  $BaFe_{12}O_{19}@MoS_2$  (C<sub>10</sub>) sample has a reflection loss of –35.41 dB (17.37 GHz) and an absorption rate of 99.97 %. The increase in thiourea on the  $BaFe_{12}O_{19}@MoS_2$  core-shell composite shows that the entire surface of the barium hexaferrite is perfectly covered with  $MoS<sub>2</sub>$  nanoflowers. This research aligns with a previous study [7]. The increase in  $MoS<sub>2</sub>$  nanoflower growth is highly validated and matches the SEM morphological results in Fig. 5, *f*.

Fig. 13 shows that an increase in hydrothermal temperature reduces the reflection loss, namely 228.47 %, from –10.78 dB (17.37 GHz) to –35.41 dB (17.37 GHz).



Fig. 13. Reflection loss of the  $BaFe_{12}O_{19}@MoS_2$  core-shell composites at various hydrothermal temperatures

Controlling the hydrothermal temperature will affect the structure and morphology of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core$ shell composite. In this research, the hydrothermal synthesis has the lowest RL and thickness at 220 °C. The precursors get enough Gibbs free energy to separate into larger particles. These findings are consistent with prior studies [28]

that have often seen an increase in temperature leading to the expansion of particle size.

Fig. 14 shows that as the hydrothermal holding time increases, the reflection loss decreases by 58 %, from –22.41 dB (13.53 GHz) to –35.41 dB (17.37 GHz).



Fig. 14. Reflection loss of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite at various hydrothermal holding times

Based on Fig. 10, the results of the PSA analysis prove that an increase in hydrothermal holding time causes an increase in particle size. Meanwhile, the SEM analysis results in Fig. 7 show that the increase in hydrothermal holding time affects the morphology and particle size of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  core-shell composite. According to a previous study [28, 29], increasing the hydrothermal holding time changes how stable the nucleation of oxide powders is and makes the nucleation happen more, which makes the particles bigger.

A previous study [30] reported that increasing the sample thickness of RAMs decreases overall reflection loss. Increasing the sample thickness of the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  core-shell composite reduces reflection loss by 69.79 %, as shown in Fig. 15. When the sample has a thickness of 2.5 mm, it has been shown that up to 99.97 % of electromagnetic waves (EM) are absorbed. Additionally, at a frequency of 17.19 GHz, the reflection loss is measured at –35.48 dB. In this study, the thinnest sample thickness of 2 mm achieved a reflection loss of –35.41 dB (at a frequency of 17.37 GHz), resulting in an EM wave absorption rate of 99.97 %, the most attractive outcome.

These findings are consistent with prior studies [31] that have often seen an increase in temperature leading to the expansion of particle size. As shown in Fig. 15, increasing sample thickness improves electromagnetic wave absorption.

Table 6 shows  $BaFe_{12}O_{19}@MoS_2$  synthesis achieving the lowest reflection at the thinnest sample thickness. It indicates that the purpose of this research is achieved.

Fig. 16 illustrates the growth mechanism of  $MoS<sub>2</sub>$ nanoflowers on  $BaFe<sub>12</sub>O<sub>19</sub>$  during the synthesis of the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite. This growth process is affected by various factors, including thiourea composition, temperature, sample thickness, and the duration of the hydrothermal synthesis.

Hydrothermal synthesis prepares a path for the growth of  $MoS<sub>2</sub>$  nanoflowers on the surface of BaFe<sub>12</sub>O<sub>19</sub>.

The concentration of thiourea directly influences the growth of the  $MoS<sub>2</sub>$  core-shell in the  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$  composite. As the thiourea composition increases, there is a corresponding increase in the growth of the  $MoS<sub>2</sub>$  core-shell. Consequently, the  $BaFe_{12}O_{19}$  $@MoS_2$  core-shell composite exhibits a higher tendency to absorb electromagnetic wave absorption to its maximum capacity.



Fig. 15. Reflection loss of the BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub> core-shell composites  $(C_{10})$  at various sample thicknesses



Fig. 16. Mechanism of  $MoS<sub>2</sub>$  nanoflower growth on  $BaFe<sub>12</sub>O<sub>19</sub>$  in the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite

Table 6

Summary of recent findings on composites with a ferrite matrix or molybdenum as reinforcement decreases the ability to reflect an electromagnetic wave

Material	RL (dB)	Frequency (GHz)	Thickness (mm)	Reference
CF@MoS <sub>2</sub>	$-21.4$	10.85	3.8	[32]
2-0 MoS <sub>2</sub> Ni NPs	$-19.7$	16.44	2.92	[33]
$MoS2@Ti3C2Tx$	$-51.0$	6.1	4.0	[34]
MoS <sub>2</sub> @AC	$-31.8$	16.72	7.0	$[35]$
Fe <sub>3</sub> O <sub>4</sub> @Fe	$-30.5$	6.2	2.0	[36]
$BaFe12O19@CaF2O4$	$-36.8$	9.9	2.9	$[37]$
$BaFe_{12x-2x}(Mn_05Co_05Ti)_xO_{19}$	$-40.2$	14.9	2.3	[38]
$BaFe12O19@MoS2$	$-35.41$	17.37	2.0	This study

#### **6. Discussion of the investigation results of BaFe12O19@ MoS2 core-shell composite by controlling hydrothermal parameters**

The study began by observing that the  $BaFe_{12}O_{19}@MoS_2$ core-shell composite single phase has appropriately been synthesized from the alloy of its raw materials, as shown in Fig. 2 and enhanced by refinement using the Material Analysis Using Diffraction (MAUD) software to make the observed pattern match the calculated ones shown in Table 2. Refinement findings were allowed if they fulfilled the R-weighted profile (Rwp), R-expected profile (Rexp), and SIG reliability index. The SIG parameter in the MAUD programme is used to assess how well crystal structure models generated from x-ray diffraction patterns corresponded to actuality. This metric assesses the similarity of observed and calculated diffraction patterns. During refinement, parameter values are expected to fluctuate. The higher the agreement between observed and calculated diffraction patterns, the lower the accuracy index. The efficiency of the refinement in this inquiry is proved by its compliance with the accuracy constraints of SIG 4, Rwp 20 %, and Rexp 15 %, as evidenced in Table 2. The EDS mapping in Fig. 3 is confirmed to have a successfully homogeneous distribution. Since the composite is a nonconductive material, the samples will be coated with gold. Therefore, Fig. 4 confirms the detection of gold as a coating during the sample preparation. The presented composition of element mapping is confirmed in Table 3. The absorption of electromagnetic waves in anti-radar composite materials is associated with the morphological shape of the composite surface. As seen in Fig. 5, 7, the morphological formation of nanoflowers using SEM proved effective in absorbing electromagnetic waves. This study also observed the effect of increasing the composition of thiourea in composites. Increasing the composition of thiourea will increase  $BaFe_{12}O_{19}@MoS_2$ particle size, as shown in Fig. 6, and the effect will improve the absorption of electromagnetic waves. As shown in Fig. 8–10, in the composite process, the composition of thiourea, temperature, and holding time can all impact the particle size distribution. Fig. 5 confirms that  $BaFe_{12}O_{19}$  has a hexagonal, plate-like structure.  $MoS<sub>2</sub>$ , on the other hand, tends to generate spherical nanoflowers even though the XRD characterization shows that they both have the same crystal structure, namely hexagonal. PSA can form spherical particles even though BHF has a hexagonal crystal structure because of differences in particle size between PSA and SEM characterization, as shown in Tables 4, 5. Simultaneously, it is essential to note that SEM analysis is subject to a limitation on the spectrum point, representing the whole surface of the sample. The intensity of light diffused on the particles in the suspension is used to determine particle size. According to dynamic light scattering (DLS), when dispersed particles are exposed to a monochromatic ray, the intensity of light dispersing on fluctuating moving particles depends on the diffusion coefficient [32–39]. The slower movement of larger particles leads to a slower fluctuation in the emitted light intensity, while smaller particles exhibit quick fluctuations, resulting in intensity oscillations. The oscillation frequency calculates the diffusion coefficient depending on the particle size [40].

According to a study [41], the addition of Mo dope caused a decrease in the values of *Ms*, *Mr*, and *Hc* when compared to the untreated samples, as shown in Fig. 11. By looking at the thiourea composition (Fig. 12), temperature (Fig. 13), hydrothermal holding time (Fig. 14), and sample thick-

ness (Fig. 15), the study was able to find different ways to reduce the reflection loss. An increase in hydrothermal temperature increases the number and growth of nanoflower cell nuclei, while an increase in hydrothermal holding time increases the density and depth of the nanoflower petals. The large  $MoS<sub>2</sub>$  nanoflower structure with tightly closed petals tends to produce maximum EM dissipation and scattering effects in core-cell composite systems. The hydrothermal temperature parameter has the most significant effectiveness in reducing reflection loss by 228.47 %, followed by the thiourea composition parameter with a reduction of reflection loss of 149.01 %, the sample thickness parameter with a reduction of reflection loss of 69.76 %, and the hydrothermal holding time parameter with a reduction of reflection loss of 58 %. Previous research has demonstrated that the hydrothermal temperature represents the essential synthesis parameter with the most significant impact [42].

As demonstrated in Table 6, this study produced the best candidate with an RL of –35.41 dB (17.37 GHz, Ku band) and 99.97 % absorption at the narrowest sample thickness of 2 mm. Several researchers have reported that a core-shell composite is a hierarchical structure that can improve RAM performance. Because of its dielectric properties,  $MoS<sub>2</sub>$  is becoming a more popular alternative because it may be used as a booster to modify polarization and reduce RL.

The study used a two-step molten salt synthesis approach, with a subsequent focus on the ongoing control of hydrothermal conditions. The primary objective of the first stage in molten salt synthesis is to decrease the solid-state's temperature from around 1423 K [43] to 1373 °C, as seen in Fig. 16. In contrast, the second step, involving molten salt, attempts to enhance the surface pathway of  $BaFe_{12}O_{19}$  in order to facilitate the growth pathway of  $MoS<sub>2</sub>$ . Fig. 16 illustrates that EM waves that hit the sample have three characteristics: absorption, transmission, and reflection. The design of this study is to maximize absorption and minimal reflection of EM waves. The microwave absorption mechanism of  $BaFe_{12}O_{19}@MoS_2$  core-shell composites is significantly complex and involves multiple stages. The magnetic and dielectric losses of the composite material reduce the microwave signal. Magnetic loss occurs due to  $BaFe<sub>12</sub>O<sub>19</sub>$ , which has high magnetic permeability and generates eddy currents in response to an applied electromagnetic field [44]. In addition, it is essential to note that  $BaFe_{12}O_{19}$  exhibits significant magneto-crystalline anisotropy, resulting in considerable reflection loss [45]. Moreover, the dielectric loss can be attributed to  $MoS<sub>2</sub>$  nanoflowers, characterized by a high dielectric constant and the ability to store energy inside an electric field [46, 47]. Dielectric loss occurs when a pulse of electromagnetic radiation passes through a material and causes the molecules inside the material to polarize. Polarization causes thermal effects, which result in energy dissipation and a reduction in the strength of the electromagnetic wave [18, 19]. In an external electromagnetic field, the delay of dipoles turning around would consume the incident microwave [44]. The mechanisms present include assistant eddy current, magnetic resonance loss, enhanced impedance matching, dielectric loss, multiple reflection and scattering effects, and interface polarization loss [47], all contributing to reducing the microwave signal further and improving the material's absorption performance.

This study's limitation is a challenging improvement due to the non-uniform size of the  $BaFe_{12}O_{19}@MoS_2$  particle, which resulted in a PDI value in the range of 0.3–0.7.

It is essential to work with solid-state and hydrothermal temperatures. The following study must strictly control the different temperatures between the furnace display and  $BaFe<sub>12</sub>O<sub>19</sub>@MoS<sub>2</sub>$ , which is critical to using a thermocouple. Further development of this research is measuring anti-radar performance at frequencies lower than 12 GHz to make an anti-radar more widely used in line with improved lifestyles and technological developments.

#### **7. Conclusions**

1. Incorporating  $MoS<sub>2</sub>$  into the  $C<sub>10</sub>$  composition effectively protects the material from electromagnetic wave radiation, showing the potential to block radar signals.  $MoS<sub>2</sub>$  successfully covered  $BaFe_{12}O_{19}$  and exhibited a uniform hexagonal shape evenly distributed on the surface. As seen in the  $T_{22}/H_{18}$  sample, the  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> core-shell composite grows steadily, creating a nanoflower shape. This nanoflower shape represents the development of targeted modifications. It can absorb all incident waves to their fullest extent, significantly reducing both transmitted and reflected waves. This study successfully achieved the single-phase crystal structure of a BaFe<sub>12</sub>O<sub>19</sub>@ MoS2 core-shell composite. In this investigation, the composite system did not affect the crystal systems of its constituent compounds. The modification of the core-shell composite tends to follow the properties of  $BaFe<sub>12</sub>O<sub>19</sub>$  as the core, demonstrating little change in the lattice parameters, the volume of the unit cell, and density.

2. The hydrothermal temperature contributes most crucially to controlling the morphology of the core-shell composite. Hydrothermal temperature control above 200 °C effectively prevents the emergence of nanorods that inhibit the absorption process of electromagnetic waves. Increased thiourea composition, temperature, and hydrothermal durability increase particle size. The core-shell composite nanoflower particle size increases with the petals' enhanced quantity and thickness to reduce reflection loss through scattering and dissipation effects. The growth and development of core-shell  $MoS<sub>2</sub>$  nanoflowers are affected by increasing hydrothermal temperature.

3. The investigation's findings demonstrated that the  $MoS<sub>2</sub> composition was the only factor influencing the mag$ netic properties of the  $BaFe_{12}O_{19}@MoS_2$  core-shell composite. The increased  $MoS_2$ -composition of the  $BaFe_{12}O_{19}$ succeeded in changing the hard magnet properties into soft magnets, which were the condition of the absorber materials.

4. A core-shell composite of  $BaFe_{12}O_{19}$ @MoS<sub>2</sub> demonstrated anti-radar capability with a low reflection loss of –35.41 at a temperature of 220 °C and a holding time of 18 hours. The remarkable of this study is that obtaining the thinnest sample thickness of 2 mm can result in microwave absorption of up to 99.97 % at a frequency of 17.37 GHz. The hydrothermal temperature is a crucial process parameter that has played an essential part in reducing reflection loss up to 228.47 %.

# **Conflict of interest**

The authors declare that they have no conflict of interest concerning this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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#### **Data availability**

Data will be made available on reasonable request.

#### **Use of artificial intelligence**

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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