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## Radar Absorption Performance of $Fe_3O_4/AC/PANI$ Nanocomposites Prepared from Natural Iron Sand

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#### PAPER INFO

#### ABSTRACT

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Keywords: Fe<sub>3</sub>O<sub>4</sub>/AC/PANI Iron Sand Nanocomposite Radar Absorbing Material Simple Coprecipitation Method In this work, the Fe<sub>3</sub>O<sub>4</sub> nanoparticles from natural iron sand were combined with active carbon (AC) and polyaniline (PANI) to obtain Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites with mass variations of the AC of 0.1, 0.2, 0.3, 0.4, and 0.5 g. The crystalline phase of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites formed from Fe<sub>3</sub>O<sub>4</sub> with PANI having an amorphous phase. Meanwhile, the crystalline phase of AC was unmatched because of its very small composition. The presence of AC was observed through vibrations from the C-C and COOH functional groups. The existence of PANI was indicated by the vibrations of the benzoic ring and quinonoid bonds. Besides, the presence of Fe<sub>3</sub>O<sub>4</sub> was confirmed by the presence of Fe<sub>-</sub>O functional groups from octahedral and tetrahedral positions. The optical properties of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites were shown by increasing the energy gap along with decreasing absorption wavelength. Interestingly, increasing AC composition made the absorption bandwidth of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites that reached -15.8 dB. The increase in the radar absorption performance of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites came from the efficient complementarity between dielectric loss and magnetic loss and interfacial polarization between Fe<sub>3</sub>O<sub>4</sub>-AC or between Fe<sub>3</sub>O<sub>4</sub>-PANI.

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NOMENCLATURE					
RAM	Radar Absorbing Material	XRD	X-ray Diffraction		
RL	Reflection Loss	IR	InfraRed		
PANI	polyaniline	SEM	Scanning Electron Microscopy		
AC	Activated Carbon				

#### **1. INTRODUCTION**

In the last decades, the applications of electromagnetic wave-based equipment in various fields have been expanded rapidly. On the other hand, the use of electromagnetic waves can also cause problems in environmental pollution, human health, electromagnetic interference, and so forth [1]. Consequently, to unravel this problem, studies related to the design and fabrication of electromagnetic wave absorbers become essential to be conducted.

One of the studies on electromagnetic wave absorbers which began to be widely studied by researchers was radar absorbing materials (RAM) [2,3]. Theoretically, RAM is an absorbent material for microwaves that works by reducing the radar cross-section or converting microwaves to heat energy [4]. It thus alleviates the reflection (*RL*) of the radar so that the pollution caused can be reduced. In addition, one of the resilient reasons why research related to RAM began to develop rapidly is because RAM can be applied to the military field to assist the defense of a country in terms of protection strategies during the war [5]. Technically, RAM coating on a fighter or warship decreases the cross-section of the radar [6], so that the reflected waves decrease or even disappear.

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In general, radar waves consist of magnetic and dielectric components in perpendicular directions [7]. Therefore, the development of RAM fabrication is worthdoing through engineering composite materials that have excellent magnetic and dielectric properties to maximize radar absorption [8]. One magnetic material that is incredibly promising for RAM is Fe<sub>3</sub>O<sub>4</sub> nanoparticles inasmuch it possesses high magnetization and permeability characteristics [9], good flexibility [10], excellent chemical and thermal stability [11]. Unfortunately, Fe<sub>3</sub>O<sub>4</sub> entails disadvantages since it has low dielectric properties, easily agglomerated [12], and high density which affects the absorption of radar waves [3]. On the other hand, RAM applications require materials that have a wide bandwidth [13] with low density [14]. Therefore, Fe<sub>3</sub>O<sub>4</sub> nanoparticles need to be modified with other materials that have dielectric properties and low density, such as with polyaniline polymers (PANI).

Based on previous works, it was observed that PANI is a polymer which has a high conductivity [1], controlled dielectricity, good environmental stability [15], high conductivity, and low density [16]. Thus, this material has extremely decent potential as RAM that is compiled with Fe<sub>3</sub>O<sub>4</sub> nanoparticles. With regard to this fact, a previous study has also succeeded in synthesizing RAM based on Fe<sub>3</sub>O<sub>4</sub>/PANI composites with reflection loss (RL) values of less than -10 dB [17]. However, for special applications that require high absorption, to refine the performance of the Fe<sub>3</sub>O<sub>4</sub>/PANI composites in radarabsorbing is needed. Therefore, to refine its absorption performance, the Fe<sub>3</sub>O<sub>4</sub> PANI composites need to be modified by adding other materials that have a wide surface and porous structure in forming more complex nanocomposites.

Based on previous studies, it is observed that activated carbon (AC) is a dielectric material that has a high absorption [18–20] since it has a large surface area and a porous structure [21]. Other research also proved that the AC/Fe<sub>3</sub>O<sub>4</sub> composites application employed as a microwave absorbing material successfully obtained a maximum RL of -10.09 dB [22]. Therefore, through the development of new fabrication materials based on the combination of Fe<sub>3</sub>O<sub>4</sub>, PANI, and AC to form the nanocomposites Fe<sub>3</sub>O<sub>4</sub>/AC/PANI proposed in this work is potential to advance the performance of radar absorption far better than before. The hypothesis that there is an increase in the performance of radar absorption through the importance of the nanocomposites application is at least predictable due to the effect of efficient complementarity between dielectric loss and magnetic loss and interfacial polarization between Fe<sub>3</sub>O<sub>4</sub> and AC or between Fe<sub>3</sub>O<sub>4</sub> and PANI.

While it is insufficient to modify the  $Fe_3O_4/AC/PANI$  nanocomposites to increase its absorption of radar waves, the development of inexpensive and simple synthesis

methods to reduce fabrication costs are encouraged. Thereby, in this work, we developed natural fabrication methods of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites in the form of iron sand using a simple precipitation method. Iron sand that is easily obtained in abundant quantities in almost all regions of Indonesia provides new opportunities in the fabrication of inexpensive and environmentally friendly Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites. While choosing the precipitation method can only be done at room temperature, the process is simple, fast, and low-cost. Furthermore, to find out the optimal performance of nanocomposite absorption, in this work, the effect of AC composition variation on the absorption of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites radar waves is examined. The different weight ratios of AC: (Fe<sub>3</sub>O<sub>4</sub>/AC/PANI) nanocomposites were of 1:30, 2:30, 3:30, 4:30, and 5:30. Nonetheless, the study of the structure, functional groups, morphology and particle size, and the optical properties of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI are also an important part of this work.

#### 2. MATERIALS AND METHODS

**2.1. Materials** The materials used in this work are Indonesian iron sand, HCl, NH<sub>4</sub>OH, H<sub>2</sub>O,  $C_6H_5NH_2$ , (NH)<sub>4</sub>S<sub>2</sub>O<sub>8</sub>, and AC.

**2. 2. Synthesis of Fe<sub>3</sub>O<sub>4</sub>/AC Nanocomposites** In this work, the Fe<sub>3</sub>O<sub>4</sub>/AC nanocomposites were fabricated using the coprecipitation method started with the preparation of FeCl<sub>2</sub> and FeCl<sub>3</sub> solutions through the reaction of the iron sand with HCl. A total of 15 ml of FeCl<sub>2</sub> and FeCl<sub>3</sub> solutions added by AC with composition variations of 0.1, 0.2, 0.3, 0.4, and 0.5 g were employed. Subsequently, the solution was stirred for 35 minutes and followed by the NH<sub>4</sub>OH titration process to obtain concentrated black deposits which were Fe<sub>3</sub>O<sub>4</sub>/AC deposits. Furthermore, the precipitate was washed by using H<sub>2</sub>O to obtain a pH = 7 and dried at 100 °C to obtain Fe<sub>3</sub>O<sub>4</sub>/AC nanocomposites.

2. 3. Synthesis of PANI PANI powder was synthesized by using the in-situ polymerization method of the C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub> monomer. A total of 2 mL of C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub> were reacted with 50 mL HCL and named as solution A. Furthermore, the  $(NH_2)_4S_2O_8$  powder of 6.72 g was added to 50 mL H<sub>2</sub>O and named as solution B. Solutions A and B were each stirred by using a magnetic stirrer at a speed of 650 rpm for 60 minutes and then this solution was allowed to stand for 1 hour followed by the stirring process by using magnetic stirrer for 120 minutes with a speed of 450 rpm which was then allowed to stand for 24 hours to obtain a dark green precipitate. To obtain PANI, the precipitate was then washed by using 50 mL HCL and followed by a washing process with H<sub>2</sub>O followed by a filtering and drying process for 60 minutes at 100 °C.

#### 2. 4. Synthesis of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI Nanocomposites

The Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites were synthesized by using a simple precipitation method. A total of 3.0 g of Fe<sub>3</sub>O<sub>4</sub>/AC with an AC mass of 0.1 g and a mass of 0.36 g PANI were dissolved in 50 mL H<sub>2</sub>O. The solution was then stirred by using a magnetic stirrer at 760 rpm for 15 minutes. Besides, the solution was filtered to obtained the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI precipitate and continued with the drying process at 100 °C for 60 minutes. The process was repeated 5 times with variations in AC mass on the Fe<sub>3</sub>O<sub>4</sub>/AC nanocomposites of 0.1, 0.2, 0.3, 0.4, and 0.5 g. Thus, in this work, there were 6 samples, these are,  $Fe_3O_4$ (coded AC-0), Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites with AC mass variations of 0.1, 0.2, 0.3, 0.4 and 0.5 g which were consecutively coded with AC-0.1, AC -0.2, AC-0.3, AC-0.4, and AC-0.5. The mechanism of formation of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites carried out in this study was illustrated in Figure 1.

**2. 5. Characterizations** The structure of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites were characterized by using X-ray diffractometer (XRD), Cu-K $\alpha$  1.540 Å. Scanning electron microscopy (SEM)-EDAX was used to observe the morphology and composition of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites constituents. The Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites functional group was characterized by an FTIR spectrometer. The optical properties of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites were obtained from the characterization by using a UV-Vis spectrometer. Furthermore, radar absorption of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites was investigated by employing a vector network analyzers (VNA).

#### **3. RESULTS AND DISCUSSION**

The X-ray diffraction patterns of AC and PANI are shown in Figure 2. In Figure 2 (a), the peak of AC diffraction is identified at position  $2\theta$  around  $23.6^\circ$ , broad peak at  $43.8^\circ$  and  $67.8^\circ$ , where all three peaks show sequential diffraction (002), (100), and (004) corresponding to the graphite lattice [23,24]. Figure 2 (b) shows the diffraction pattern of PANI with a broad amorphous peak at position  $2\theta$  about 25.3° of the plane (200). The emergence of the broad amorphous peak shows the existence of parallel and repeated polymer chains and yields information if PANI is in the form of



Figure 1. Formation mechanism of  $Fe_3O_4/AC/PANI$  nanocomposites

emeraldine salt [25]. Furthermore, Figure 3 indicates the results of the XRD characterization of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites. Phase analysis of the nanocomposites were carried out by comparing the diffraction patterns of the characterization results with the ICSD-30860 model data, which identified several peaks at positions  $2\theta 30.1^{\circ}$ (002), 35.5° (113), 43.2° (004), 47.3° (133), 53.6° (224),  $57.1^{\circ}$  (115) and  $62.7^{\circ}$  (044). These peaks confirm that the nanocomposites are only composed of one crystalline phase of Fe<sub>3</sub>O<sub>4</sub>. The absence of AC peak is caused by the mass of AC which tends to be small when compared to Fe<sub>3</sub>O<sub>4</sub> in the composites. Meanwhile, the emergence of PANI peak is caused by this polymer having an amorphous structure. Based on the results of the phase analysis, Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites have an inverse cubic spinel structure with a = b = c values ranging from 8.366 to 8.382 Å which correspond to the results of previous research [15].

The particle size of the  $Fe_3O_4/AC/PANI$  nanocomposites was analyzed quantitatively by using the Scherrer equation shown in Equation (1).

$$D = \frac{K.\lambda}{\beta.\cos\theta} \tag{1}$$

where *D* is the particle size (nm), *K* is a constant (0.94),  $\lambda$  is the wavelength (1.5443 Å),  $\beta$  is the full width of halfmaximum, and  $\theta$  is the Bragg angle from the highest peak [2]. The particle sizes obtained from the fitting results by using the Lorentzian method are shown in Table 1.



Figure 2. X-ray diffraction patterns of (a) AC and (b) PANI



Figure 3. XRD patterns of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites

TABLE 1. Particle size of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocompositesSamples $2\theta$  at the highest peak (°)Particle Size (nm)

AC-0	35.4	9.87
AC-0.1	35.6	19.49
AC-0.2	35.5	21.49
AC-0.3	35.6	25.72
AC-0.4	35.5	16.90
AC-0.5	35.5	24.74

In general, the addition of the composition of AC and PANI to  $Fe_3O_4$  nanoparticles causes the crystallinity level of  $Fe_3O_4$  to increase. This is proven in Figure 3 which shows that the peaks of  $Fe_3O_4$  in nanocomposites (AC-0 to AC-0.5) appear sharper when compared to pure  $Fe_3O_4$ . The increase in AC composition in  $Fe_3O_4/AC/PANI$  nanocomposites results in an increase in the intensity of the  $Fe_3O_4$  peak except for AC-0.4 which is lower when compared to AC-0.3. This is because the AC-0.4 particle size is lower than other nanocomposites of particle sizes. This is also confirmed in Table 1 which shows the particle size of the sample.

Figure 4 portrays the FTIR spectrum of PANI, AC, Fe<sub>3</sub>O<sub>4</sub> nanoparticles and Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites. The spectrum of the PANI sample indicates vibrational peaks at wavenumbers 825, 1184, 1336, 1504, and 1587 cm<sup>-1</sup>. Meanwhile, the vibrations observed in the wavenumber of 825 cm<sup>-1</sup> shows the C-H vibrations. These results seem similar to the results of previous studies that detected C-H vibrations at wavenumbers of 812 cm<sup>-1</sup> [26]. The functional group of N = Q = N is detected at a wavenumber of 1184 cm<sup>-1</sup>, where Q is a quinoid [27] which, at a wavenumber of 1336 cm<sup>-1</sup>, is the vibration of C-N. The strongest vibrational peaks were detected at wavenumbers of 1504 and 1587 cm<sup>-1</sup> which are related to the functional groups of C = C quinoids and benzoic. The intensity of transmittance in the quinoid and benzoic groups has the same value, confirming that PANI is in the form of



Figure 4. IR spectrum of AC-0, PANI, and AC

emeraldine salt. In addition to the 5 main vibrations from PANI, vibrations in the region from 3400 to 3500 cm<sup>-1</sup> were also detected, indicating vibrations from N-H which originates from the aromatic amine functional group. This was also confirmed by previous studies that detected aromatic amine functional groups at wavenumbers of 3500 cm<sup>-1</sup> [27].

Pure AC samples shown in the FTIR spectrum show vibrations from C = C, C = O, and O-H from the carboxyl group and C-O stretching derived from the acid used to activate carbon. Sequentially, these vibrations were detected at wavenumbers of 1592, 1702, 3200, and 1032 cm<sup>-1</sup>. And the results correspond to the previous studies [28]. Meanwhile, the Fe<sub>3</sub>O<sub>4</sub> nanoparticles depicted by AC-0 spectrum several main vibrations were detected in the form of Fe-O at a wavenumber of 418 cm<sup>-1</sup> and in the region of 541-651 cm<sup>-1</sup>, which sequentially showed octahedral and tetrahedral Fe-O vibrations. This confirms the XRD results stating that Fe<sub>3</sub>O<sub>4</sub> has an inverse spinel cubic structure composed of octahedral and tetrahedral crystal systems. These results correspond to the previous studies that showed Fe-O vibrations at 417 cm<sup>-1</sup> [29] and 580-590 cm<sup>-1</sup> [30]. Interestingly, after Fe<sub>3</sub>O<sub>4</sub> was composited with AC and PANI in AC-0.1 to AC-0.5 samples, it was observed that the transmittance intensity of the Fe-O functional groups decreased with the increase in AC composition. This confirms that the Fe<sub>3</sub>O<sub>4</sub> mass ratio has decreased after it was compiled with AC and PANI. The visible shift of vibration peaks from PANI after being composed of Fe<sub>3</sub>O<sub>4</sub> and AC nanoparticles. Vibration N = Q = N was detected at a wavenumber of 1153 cm<sup>-1</sup>, C-N at a wavenumber of 1315 cm<sup>-1</sup>, and C = C at a wavenumber of 1508 and 1591 cm<sup>-1</sup>. Furthermore, the phenomenon of decreasing the intensity of transmittance at 3200 cm<sup>-1</sup> wave number shows the O-H functional group. This happens because O-H is a carboxyl functional group with a negative charge so that when composited with positively charged Fe-O, it will bind to one another.

SEM The images of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites with an AC composition of 0.3 g is shown in Figure 5. As shown in Figure 5 (a), the morphology of the nanocomposites consists of 3 particle shapes namely spheres, chunks, and worm-like structures. Sequentially, these shapes describe the Fe<sub>3</sub>O<sub>4</sub> nanoparticles, AC [31,32], and PANI [33]. Qualitatively, it is observed that the sphere of Fe<sub>3</sub>O<sub>4</sub> tends to agglomerate. The size of Fe<sub>3</sub>O<sub>4</sub> nanoparticles is smaller than that of AC which has relatively irregular chunks. In addition, the AC surface also shows some agglomeration of Fe<sub>3</sub>O<sub>4</sub>, while PANI fills the gap between Fe<sub>3</sub>O<sub>4</sub> and AC [27]. Illustrated in Figure 1, AC is a carbon material that is activated with acids to form OH-functional groups, so that the AC will bind to the positive charge of Fe<sub>3</sub>O<sub>4</sub> which causes the  $Fe_3O_4$  to fill the surface of the AC. Based on Figure 5(a), few AC surfaces are covered by



**Figure 5.** SEM images of AC-0.3 with (a) 5000 and (b) 200,000 magnifications, and (c) particles size distribution

agglomeration of Fe<sub>3</sub>O<sub>4</sub>. This is because the density of Fe<sub>3</sub>O<sub>4</sub> is higher than that of AC [22]. Figure 5(b) indicates the morphology of Fe<sub>3</sub>O<sub>4</sub> nanoparticles with a magnification of 200,000 magnification and shows that the Fe<sub>3</sub>O<sub>4</sub> nanoparticles are evenly dispersed on the AC surface. Based on the results of data analysis in Figure 5(c), it is observed that the particle size distribution of Fe<sub>3</sub>O<sub>4</sub> is 21.5 nm. This approximates the particle size values of the XRD results calculated in Table 1. Furthermore, the EDAX results show that the composition of heavy elements C, O, and Fe are 28.17, 19.63, and 52.20%, respectively, which confirms the presence of Fe<sub>3</sub>O<sub>4</sub> nanoparticles, AC, and PANI that form Fe<sub>3</sub>O<sub>4</sub>/AC/PANI composites. The presence of Fe<sub>3</sub>O<sub>4</sub> was confirmed by the presence of Fe and O elements. Meanwhile, the AC and PANI were confirmed by the presence of element C, which is carbon as the main element of AC and the benzoic and quinoid ring bonds of PANI.

The optical properties of Fe<sub>3</sub>O<sub>4</sub>, AC, PANI, and Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites with variations in AC composition were characterized by using UV-Vis at a wavelength between 350-700 nm. Figure 6 shows the UV-Vis spectrum of Fe<sub>3</sub>O<sub>4</sub>, PANI, and AC samples. The Fe<sub>3</sub>O<sub>4</sub> nanoparticle spectrum shows absorption at a wavelength of 351 nm which shows the characteristic of Fe ions located at the octahedral site. This is in accordance with previous research stating if the absorption of Fe<sub>3</sub>O<sub>4</sub> occurs in the range of 300-750 nm [34]. Qualitatively, it is observed that PANI has the highest absorption value compared to Fe<sub>3</sub>O<sub>4</sub> and AC nanoparticles. Based on UV-Vis results, two absorption peaks in PANI were detected at the position of 355 and 512 nm. The peak at a wavelength of 355 nm indicates the  $\pi$ - $\pi$ \* transition, while the absorption at 512 nm indicates the  $\pi$ - $\pi$ \* transition from the benzoic ring to the quinoid ring.

The absorbance spectrum of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites with variations in the AC mass composition is illustrated in Figure 7. Based on the figure, the absorption at wavelengths ranging from 510.7 nm to 514.5 nm is shown in Table 2. When compared absorption at wavenumbers around 512 nm (Figure 6) is found, the range of wave numbers shown in Figure 6 is wider with higher absorption. That is because the absorption at that wavelength is a combination of absorption by AC and PANI. Prior to being compiled with Fe<sub>3</sub>O<sub>4</sub> and AC, a benzoic ring transition was detected at 512 nm, and after it was made, a transition wavelength shift occurred. Theoretically, this is due to an increase in orbital antibonding energy, which is due to the interaction between PANI and Fe<sub>3</sub>O<sub>4</sub>/AC. Such results were confirmed by a previous work of a shift in absorption peaks from PANI as a polymer after being combined with Fe<sub>3</sub>O<sub>4</sub> nanoparticles [1].

Theoretically, electrons in the outer shell of an atom absorbing radiation energy shift to a level with higher energy or called electron excitation [35]. Thus, the absorbance of the nanocomposite is related to the energy used by electrons to move from the valence band to the conduction band (excitation) called the energy gap [36]. The direct transition of electrons written in Equation (2) was used to calculate the energy gap of the samples.

$$(\alpha hv) = A(hv - E_g)^2 \tag{2}$$



Figure 6. Absorbance of the AC-0, PANI, and AC



**Figure 7.** Absorbance of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites

where  $\alpha$  is the absorption coefficient, *A* is the effective mass of the electron, *h* is the Planck constant which is  $6.55 \times 10^{-34}$  Js, and *v* is the frequency [35]. The energy gap values were analyzed by using the Tauc plot method, where the energy gap is an intercept on the *x*-axis of the graph of the relationship between hv and  $(\alpha hv)^2$ .

The results of the fitting by using the Tauc plot method to obtain the energy gap values are shown in Figure 8. The energy gap value of the Fe<sub>3</sub>O<sub>4</sub> nanoparticles is shown in Figure 8 with the AC-0 code of 3.307 eV. This result is similar to a previous study that obtained an energy gap value of Fe<sub>3</sub>O<sub>4</sub> of 2.87 eV [37]. In the AC and PANI materials, the energy gap values are 3.342 and 3.302 eV, respectively. These are also consistent with previous studies showing that AC has an energy gap value of less than 4.0 eV [38] and approximately 3.0 eV for PANI [39].

The energy of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI gap nanocomposites is shown in Table 2. The increase in the energy gap of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites is influenced by the mass of the AC, whereas the AC composition increases, the energy gap value also augment. The difference in the energy gap value between Fe<sub>3</sub>O<sub>4</sub> nanoparticles and Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites is influenced by particle size. Based on the calculations by using Equation (1), the particle size of Fe<sub>3</sub>O<sub>4</sub> nanoparticles is 9.87 nm which is smaller than the particle size of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites. This causes the energy gap value of Fe<sub>3</sub>O<sub>4</sub> to be greater than the nanocomposites as the effect of the particle size of the samples. Theoretically, the particle size is inversely proportional to the particle size of the material [37,40]. In addition, the energy gap value is also influenced by the absorption wavelength, where the higher the wavelength, the lower the energy gap value is. The relationship between the energy gap and absorption wavelength values in this study are shown in Table 2.

Reflection loss is a characteristic that shows the ability of radar wave absorption from  $Fe_3O_4/AC/PANI$  nanocomposites. Figure 9 shows *RL* of the  $Fe_3O_4/AC/PANI$  nanocomposites at a frequency of

TABLE 2. Energy gap of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites

Samples	Wavelength (nm)	Energy gap (eV)
Fe <sub>3</sub> O <sub>4</sub>	351.0	3.307
AC-0.1	514.2	3.100
AC-0.2	514.5	3.076
AC-0.3	512.2	3.134
AC-0.4	513.2	3.110
AC-0.5	510.7	3.175
PANI	355.0	3.342
AC	510.0	3.302



8-12 GHz which were calculated by using Equations (3) and (4).

$$RL = 20\log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|$$
(3)

where  $Z_{in}$  value was calculated with Equation (4):

$$Z_{in} = Z_0 \sqrt{\mu_r \varepsilon_r} \tanh\left[j \frac{2\pi f d}{c} \sqrt{\mu_r \varepsilon_r}\right]$$
(4)

where  $Z_{in}$  and  $Z_{in}$  are input and output impedance values. While  $\mu_r$  and  $\mathcal{E}_r$  are the permeability and complex permittivity of the material, *c* represents the velocity of the radar in a vacuum, *f* is the frequency of radar, and *d* is the thickness of the material when tested [41].

Fe<sub>3</sub>O<sub>4</sub> has an *RL* of -5.58 dB at a frequency of 10.58 GHz. The low *RL* value owned by Fe<sub>3</sub>O<sub>4</sub> is due to the fact that this sample only consists of magnetic material, thus RAM only absorbs the magnetic part of the radar. In this study, the increase in radar absorption is increased by



Figure 9. Reflection loss of the Fe\_3O\_4/AC/PANI nanocomposites

compiling Fe<sub>3</sub>O<sub>4</sub> with AC and PANI materials. That is because AC belongs to good dielectric material and PANI is classified as a conductive polymer. Based on Figure 9, it is noticed that the absorption capability of radar from Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites is stronger than that of Fe<sub>3</sub>O<sub>4</sub> nanoparticles. This indicates that the absorption properties of radar increase if added by AC and PANI materials. Physically, this phenomenon occurs since the absorption of radar from Fe<sub>3</sub>O<sub>4</sub> nanoparticles only comes from natural resonance and domain wall resonance, so that with the addition of PANI and AC, the dielectric loss properties of the two materials are introduced and the interface polarization between Fe<sub>3</sub>O<sub>4</sub> and AC or Fe<sub>3</sub>O<sub>4</sub> with PANI is introduced. In detail, the RL values of nanocomposites are shown in Table 3. Based on Table 3, it can be seen that the RL values of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites are higher than those of MWCNT/Fe<sub>3</sub>O<sub>4</sub> [42] and GN-pFe<sub>3</sub>O<sub>4</sub>@ZnO [43]. The addition of dielectric material also causes the absorption bandwidth of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites to widen. If in Fe<sub>3</sub>O<sub>4</sub> nanoparticles, the absorption bandwidth is 1.02 GHz, then when the composite bandwidth of the absorption is done, it increases within the composition of AC, which is 1.26, 1.38, 1.58, 1.68, and 1.74 GHz, respectively.

**TABLE 3.** RL value of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites

Samples	RL (dB)
AC-0	-5.58
AC-0.1	-13.09
AC-0.2	-15.00
AC-0.3	-14.06
AC-0.4	-15.80
AC-0.5	-13.99
AC@Fe <sub>3</sub> O <sub>4</sub>	-8.99 [22]
MWCNT/Fe <sub>3</sub> O <sub>4</sub>	-10.0 [42]
GN-pFe <sub>3</sub> O <sub>4</sub> @ZnO	<-10 [43]

Based on the results of the previous research, it was shown that, if the *RL* value obtained <-15 dB, it indicates that if 96.9% of the radar waves are absorbed by the nanocomposite. Meanwhile, if the RL value obtained <-20 dB, then the absorbed wave is almost 99.0% [44]. In this research, the RL value of nanocomposites has a range between -13 to -16 dB or <-20 dB. Thus, it can be concluded that the waves absorbed by nanocomposites in this study are around 96.9 - 99.0%. Two main aspects affecting the increase of radar absorption by Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites in this study are first, the efficient complementarity between dielectric loss and magnetic loss which is indicated by the relative permittivity and permeability values must be fulfilled [43,45]. If Fe<sub>3</sub>O<sub>4</sub> nanoparticles stand alone as a compiler of RAM, it can be construed that the resulting RL value is still low. This is due to the large disparity between permeability and permittivity which interferes with impedance matching. Thus, in the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI system, the Fe<sub>3</sub>O<sub>4</sub> nanoparticles act as absorbents of the magnetic parts, and PANI, as well as AC, are as dielectric absorbers of incoming radar. Besides, the AC also acts as a nucleation site for Fe<sub>3</sub>O<sub>4</sub> which prevents or reduces the aggregation of these nanoparticles. Second, there is interfacial polarization between Fe<sub>3</sub>O<sub>4</sub>-AC or between Fe<sub>3</sub>O<sub>4</sub>-PANI, where multi-interfaces on nanocomposites will produce significant polarization interfaces that will increase the value of dielectric loss at high frequencies [41]. This is indicated when the addition of AC and PANI absorption material occurs at a frequency of around 11 GHz. Thus, the development of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites in this research provides new opportunities for large-scale development for the application of high-performance microwave absorbing materials based on local natural materials through environmentally friendly synthesis.

#### 4. CONCLUSION

The Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites were successfully synthesized by using an environmental precipitation method. The presence of Fe<sub>3</sub>O<sub>4</sub> is detected as crystalline with a cubic structure. Meanwhile, the presence of AC and PANI is confirmed by C-C bond, COOH vibrations, benzoic ring vibration, and quinonoid bond. AC acts as a nucleation site for Fe<sub>3</sub>O<sub>4</sub> and PANI acts as a connector. The energy gap of Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites ranges from 3.000 to 3.175 eV. With the addition of AC and PANI in the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites, their *RL* increases significantly because the nanocomposites consist of magnetic loss and dielectric loss which increases the radar absorption. Interestingly, the RL value of the Fe<sub>3</sub>O<sub>4</sub>/AC/PANI nanocomposites ranges from -13.0 to -15.8 dB which shows their radar absorption capability is in the range of 96.9% - 99.0%.

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# Radar Absorption Performance of $Fe_3O_4/AC/PANI$ Nanocomposites Prepared from Natural Iron Sand

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