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## Synthesis and Microwave Absorption Properties of Ni<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>/CI Composite Coated with Polyaniline within Paraffin Wax Matrix

Ternary composites of polyaniline/Ni<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>/carbonyl iron (PANI/F/CI) are prepared via two stages. Firstly, Ni<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub> is prepared using a sol-gel method. After that, PANI/F/CI composites are prepared using an in-situ polymerization technique of PANI in the existence of the Ni<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub> and CI. X-ray diffractometry (XRD), Fourier transform infrared (FTIR) spectroscopy, Ultraviolet-visible (UV-vis) spectroscopy, and Thermogravimetric analysis (TGA) are utilized to characterize samples. The morphology of the powders is investigated by Scanning electron microscope (SEM). The electromagnetic interference (EMI) shielding and microwave absorption (MA) properties are measured in the frequency band of 8.8–12 GHz to investigate the microwave characterization. The results refer those microwave absorption properties are related to the absorber thickness and the loading ratio of the absorber within a paraffin matrix. Minimal reflection loss of –30.8 dB at the matching frequency ( $f_m$ ) of 10.3 GHz and the absorption bandwidth under –10 dB ( $BW_{-10dB}$ ) of 2.8 GHz for 3.4 mm thickness with a surface density (SD) of 3.38 kg/m<sup>2</sup> are noticed for the PANI/F/CI composite sample. The maximum shielding efficiency (SE) of 30.12 dB at 11.0 GHz for 3.2 mm thickness is observed for the PANI/F/CI composite sample.

**Keywords:** polyaniline, carbonyl iron, composites, lightweight microwave absorber, reflection loss, absorption bandwidth, shielding efficiency, matching frequency.

### Introduction

Recent environmental pollution issues are appearing because of the quick evolution of electronic devices, involving smartphones, laptops, and intelligent devices. Electronic apparatuses emit undesirable EM waves, generating electromagnetic interference between various electronic apparatuses with a negative effect on their performance. Consequently, the disposal of EM waves resulting from EMI effectively is so important both for public protection security and electronic safety. Generally, there are two kinds of materials to absorb EM waves: firstly, magnetic loss materials such as hexagonal ferrites, spinel ferrites, and carbonyl iron, secondly, dielectric loss materials such as conductive polymers (e.g., polyaniline, polypyrrole) and carbonaceous materials (e.g., carbon black, activated carbon, carbon fibers, graphene) which have played a significant role for high-frequency EM wave absorption. Nevertheless, the drawbacks involving elevated density, low reflection absorption, and narrow wideband have hugely limited conventional loss materials' workable benefits for EM wave absorption [1, 2]. In recent years, microwave absorption composites based on polyaniline, ferrite, and carbonyl iron have obtained significant attention due to their excellent electrical and ferrimagnetic characteristics. Polyaniline-based composites have pulled in major attention for microwave absorption lately. Polyaniline is usually used to fit the requirements of high-effective microwave attenuation materials because of its superior characteristics, for example, low density, high permittivity, unique electronic conductivity, etc. Polyaniline has a unique place in the band of elevated-frequency microwave absorption materials (MAMs). Furthermore, spinel ferrites and carbonyl iron have excellent MA characteristics due to their unique magnetic characteristics. NiZn ferrites and carbonyl iron are considered suitable materials for high-frequency implementations [3, 4]. When NiZn ferrite and carbonyl iron are mixed with Polyaniline, the MA characteristics of the resultant composite are anticipated to enhance. According to this, PANI/NiZn ferrite microwave absorbers in the frequency range of 2–40 GHz were successfully prepared by Ting et al. [3].

The absorbers are prepared by dispersing PANI/NiZn ferrite nanocomposites with a weight ratio of 67% w/w within an epoxy resin matrix. The results indicated that by increasing polyaniline content in NiZn ferrite, a wide absorption frequency range could be obtained. Didehban et al. have designed microwave absorbers in the frequency band of 8–12 GHz based on NiZn ferrite-PANI (35:65) nanocomposites. The absorbers are formed by dispersing PANI/NiZn ferrite within an epoxy resin matrix of 20 w/w. The results have shown that the absorber had a  $RL_{\min}$  of  $-20$  dB at 9.1 GHz and the absorption  $BW_{-10\text{dB}}$  was 0.5 GHz for 2 mm thickness [5]. Wang et al. have designed absorbers in the frequency band of 2–8 GHz based on NiZn Ferrite/PANI (1:3) nanocomposites. The absorbers are formed by dispersing PANI/NiZn ferrite within a paraffin matrix of 75 w/w. The absorbers were prepared by the in-situ polymerization method. The results displayed that  $RL_{\min}$  was  $-32$  dB at 9.5 GHz and the absorption  $B.W_{-10\text{dB}}$  was 3.8 GHz [6]. Wang et al. have reported the MA properties of a one-dimensional uniform PANI/NiZn ferrite hybrid nanorods within a paraffin matrix of 70 % w/w in the frequency range of 2–18 GHz. They have found that the absorbers had broadband, and minimal reflection loss, where the results have indicated that the absorber had a  $RL_{\min}$  of  $-27.5$  dB at 6.2 GHz and the absorption  $BW_{-10\text{dB}}$  was 3 GHz for 2 mm thickness [7]. Wang et al. have designed absorbers in the frequency band of 2–18 GHz based on NiZn Ferrite/PANI nanocomposites. The absorbers were prepared by hydrothermal method. The results displayed that  $RL_{\min}$  was  $-17$  dB at 11.1 GHz and the absorption  $B.W_{-10\text{dB}}$  was 5 GHz [8]. Ma et al. have reported the MA properties of PANI/ $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanocomposite. The MA properties were studied in the 8.2-26.5 GHz range. The absorbers were synthesized by the in-situ polymerization technique. They have found that the absorbers had broadband, and minimal reflection loss, where the results have indicated that the absorber had a  $RL_{\min}$  of  $-39.9$  dB at 22.4 GHz and the absorption  $BW_{-10\text{dB}}$  was 5 GHz for 2 mm thickness [9].

The aim of the study is to design lightweight and wide band absorbers with loading ratios not exceeding 35% with enhanced RL based on PANI/F/CI nanocomposites. The ferrite is prepared by the sol-gel method. Then, the aniline monomer is polymerized by the in-situ polymerization technique in the presence of NiZn ferrite and carbonyl iron. The prepared samples are characterized by XRD, FTIR spectroscopy, UV-vis spectroscopy and TGA. The morphologies of the ferrite and its nanocomposites are identified using SEM. The EMI shielding and MA properties are studied by measuring the minimal reflection loss, absorption bandwidth under  $-10$  dB, and shielding efficiency of the absorbers in the frequency band of 8.8–12 GHz to achieve functional characterization. To the best of the authors' knowledge, the optimization of the performance of the current PANI/F/CI nanocomposites in terms of lightweight and wide bandwidth has not been reported before.

### Experimental

**Chemicals:** Sodium dodecyl sulfate (SDS, 92.2 % purity), ammonium persulfate (APS, 95.3 % purity), nickel (II) nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 98.3 % purity), zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 98.7% purity), and iron (III) nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , 98.2 % purity) were purchased from TRADING COMPANY ANT, Russia. As well, Aniline monomer ( $\text{C}_6\text{H}_5\text{NH}_2$ , 99.5 % purity), Ammonium hydroxide ( $\text{NH}_4\text{OH}$ , 99.8 % purity), Citric acid ( $\text{C}_6\text{H}_8\text{O}_7$ , 99.0% purity) were purchased from Sigma Aldrich Company, Germany. On the other hand, carbonyl iron (CI, 99.6% purity) was purchased from Cabot Norit Company, Netherland.

**Instruments used:** A powder X-ray diffractometer (XRD, Rigaku Miniflex 600, Cu-Ka) is utilized for defining the crystal structures of the powders. Fourier Transform IR (FTIR) spectra are recorded on a Perkin Elmer spectrum 65 FTIR spectrometer in the range of 400–4000  $\text{cm}^{-1}$ . The UV-vis absorption spectra of the samples (dispersed in dimethylformamide (DMF)) are recorded using the LAMBDA 365 UV-vis spectrophotometer in the range of 250–900 nm. Thermogravimetric analysis (TGA) is done utilizing a thermal analyzer (NETZSCH 449F3A-0372-M) under a nitrogen atmosphere, from room temperature to 1000 °C under constant heating rate of 10 °C/min. A scanning electron microscope (FEI Quanta 200 3D) is utilized for defining the morphology of the powders. Finally, energy-dispersive X-ray spectroscopy (EDX, Quanta 200 3D) is utilized to know the chemical composition of prepared samples.

The microwave absorption properties of the prepared samples are calculated by using the horn antenna connected to an oscilloscope (AKTAKOM ADS-2221M).

#### Methodology

##### 1. Preparation of PANI/F/CI

Ferrite ( $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ) nanoparticles were prepared by a sol-gel method as illustrated in the following literature [10–14]. On the other hand, carbonyl iron powder was milled for 12 h at 300 rpm via the grinding

balls to obtain fine powders. NiZn ferrite and carbonyl iron were coated with polyaniline via the in-situ polymerization technique. Firstly, 6 g (90 % F and 10 % CI) was added to 100 ml distilled water under mechanical stirring at a speed of 250 rpm for 30 minutes. 3 g sodium dodecyl sulfate (SDS) and aniline were added to the solution while keeping mechanical stirring for 1 h. After that, 1 M HCl solution 80 mL was added to the solution under stirring for 1 h. Finally, 8.5 g APS was dissolved in 100 ml of an aqueous solution which was utilized as an oxidizing agent and added slowly dropwise into the solution to start the polymerization. The polymerization was allowed to proceed for 6 h with stirring in an ice bath. The resulting composite was filtered and washed many times with distilled water and ethanol, and then dried for 8 h in the furnace at 70 °C. The weight ratio of aniline/(F-CI) (1/1) was synthesized. Pure polyaniline was synthesized in a similar way but without NiZn ferrite and carbonyl iron solution for comparison purposes.

## 2. Preparation of samples for measuring the MA and EMI shielding properties

Microwave absorption and electromagnetic interference shielding properties of the samples were estimated with the free-space technique. According to this, 30–35 % w/w of the coated composites were dispersed in a paraffin wax matrix by heating and stirring for 15 min. Thereafter, the single-layer samples were molded to the dimensions of 100×100 mm to measure RL and SE in the frequency band of 8.8–12 GHz.

## Results and Discussion

### XRD patterns

Figure 1 demonstrates the XRD patterns of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI/F/CI composite and PANI. For the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  pattern, six diffraction peaks are noticed at  $2\theta$  values of  $30.04^\circ$ ,  $35.44^\circ$ ,  $43.12^\circ$ ,  $53.68^\circ$ ,  $57.18^\circ$ , and  $62.14^\circ$ , which conforms to (hkl) planes of (220), (311), (400), (422), (511) and (440), respectively. The ideal spinel structure is noticed by the peaks of NiZn ferrite [15]. The XRD pattern of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  is matched with the reference XRD patterns (JCPDS, PDF no. 08–0234). The size of the NiZn ferrite grain ( $2\theta = 35.44^\circ$ ) has been evaluated with Scherrer's equation,  $D = 0.9 \lambda / \beta \cos\theta$ , where  $D$  is the crystallite size (nm),  $\lambda$  is the X-ray wavelength,  $\beta$  is the bandwidth at half-height, and  $\theta$  is the diffraction angle in degree. The calculated crystallite size of the NiZn ferrite is 27.6 nm. On the other hand, for the carbonyl iron pattern, three characteristic peaks are noticed at  $2\theta$  values of  $44.61^\circ$ ,  $64.92^\circ$  and  $82.33^\circ$ , which conform to (hkl) planes of (100), (200), and (211), respectively. The XRD pattern of carbonyl iron resembles crystallites in which the sample mainly contains  $\alpha$ -Fe phase [16]. All the observed peaks of CI are matched with the standard XRD pattern (JCPDS, PDF no. 06-0696). The characteristic peaks of the PANI/F/CI composite show matching the characteristic peaks of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  as mentioned above. The XRD pattern of the pure PANI (Figure 1) displays an amorphous structure with two characteristic peaks at  $20.22^\circ$  and  $25.36^\circ$  which are attributed to the periodicity parallel to the polymer chains of PANI [17,18]. The XRD patterns of the PANI/F/CI composite (Figure 1) display crystalline peaks because of the existence of NiZn ferrite in this composite. The two characteristic peaks of the PANI disappeared due to the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles [19, 20].

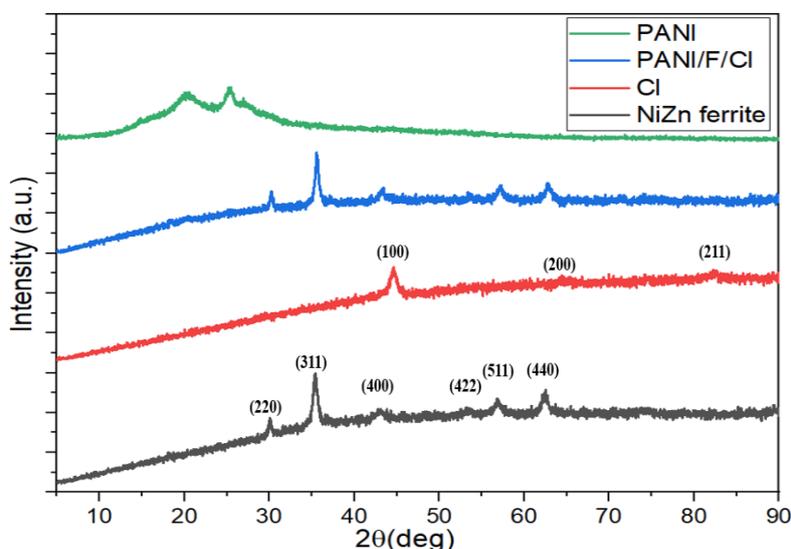


Figure 1. XRD patterns of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI/F/CI composite and pure PANI

### FTIR spectra

Figure 2 shows the FTIR spectra of the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI/F/CI composite and PANI. For the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles, two peaks at  $563.1\text{ cm}^{-1}$  and  $430.2\text{ cm}^{-1}$  are referring to the stretching vibration of (Fe–O), which emphasizes the formation of the metal-oxygen in ferrite-based [21]. In addition to that, the peak at  $1630.4\text{ cm}^{-1}$  in  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , and CI is referring to C=O stretching vibration, and the peaks at  $2348\text{ cm}^{-1}$  and  $3452\text{ cm}^{-1}$  are referring to O–H stretching vibration [22, 23]. On the other hand, the characteristic peaks of PANI and PANI/F/CI composite are similar and they exhibited peaks at  $1568\text{ cm}^{-1}$ ,  $1489\text{ cm}^{-1}$ ,  $1298\text{ cm}^{-1}$ ,  $1238\text{ cm}^{-1}$ ,  $1113\text{ cm}^{-1}$ , and  $800\text{ cm}^{-1}$  [24, 25]. The characteristic peaks at  $1568$  and  $1489\text{ cm}^{-1}$  are attributed to the C=N and C=C stretching modes of vibration for the quinonoid and benzenoid units of the polymer. The characteristic peaks at  $1298$  and  $1238\text{ cm}^{-1}$  are related to N–H bending and asymmetric C–H stretching of the benzenoid ring, respectively. Finally, the characteristic peaks at  $1113\text{ cm}^{-1}$  and  $800\text{ cm}^{-1}$  are ascribed to the vibration mode of N=Q=N and the out-of-plane stretching vibration of C–H, respectively [19, 20]. In addition to that, the characteristic peak at  $563.1\text{ cm}^{-1}$  of PANI/F/CI composite shows matching the characteristic peak of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  as mentioned above. This indicates the stretching vibration of (Fe–O), which confirms the formation of the metal-oxygen in PANI/F/CI.

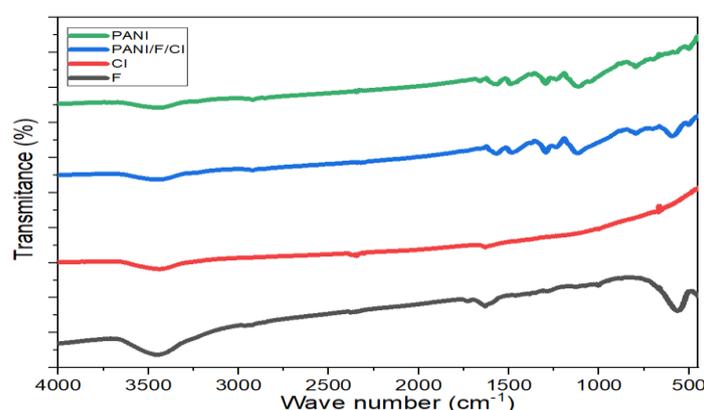


Figure 2. FTIR spectra of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI/F/CI composite and pure PANI

### UV-visible spectra

Figure 3 illustrates the UV–visible spectrum of the PANI and PANI/F/CI composite. For PANI, two characteristic peaks at around  $302\text{ nm}$  and  $629\text{ nm}$  are observed. The characteristic peak around  $302\text{ nm}$  is ascribed to  $\pi\text{--}\pi^*$  transition of the benzenoid ring and the characteristic peak around  $629\text{ nm}$  is attributed to the benzenoid-to-quinoid excitonic transition [17, 26]. It can be seen that the characteristic peaks of PANI/F/CI composite show a clear red shift of  $7\text{ nm}$ , as compared with that of polyaniline. The two characteristic peaks show the presence of PANI on the surface of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  and carbonyl iron. These results may refer to the  $\sigma\text{--}\pi$  interaction among  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , carbonyl iron and polyaniline backbone, which leads to the energy of the antibonding orbital decrease, the energy of the  $\pi\text{--}\pi^*$  transition of the benzenoid and quinoid ring decreases, so the characteristic peaks of the composite show a red shift [26].

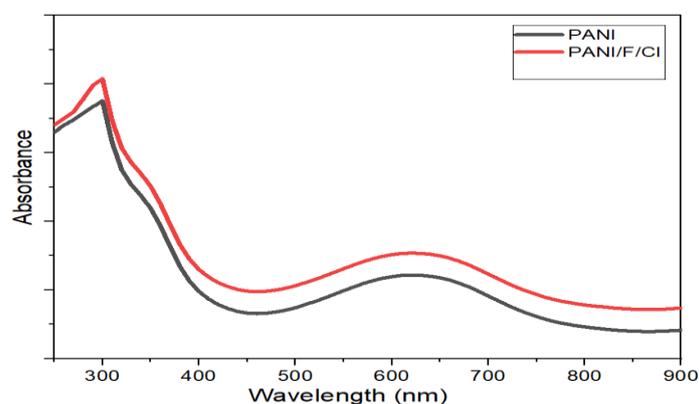


Figure 3. UV spectra of PANI and PANI/F/CI composite.

### TGA analysis

Figure 4 shows the TGA curves of the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , PANI/F/CI composite and PANI. For the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles, no mass loss was noticed over the whole temperature range. PANI loses 4.87 % of its weight in the range of 110–130 °C because of the evaporation of moisture in the PANI. The thermal decomposition of the PANI is shown in the range of 230–1000 °C and has a big weight loss of 65.12 %. On the other hand, PANI/F/CI composite loses about 2.21 % of its weight in the range of 110–130 °C which is due to the evaporation of moisture in the composite. The thermal decomposition of the PANI/F/CI composite is shown in the range of 240–870 °C and has a big weight loss of 46.28 %.

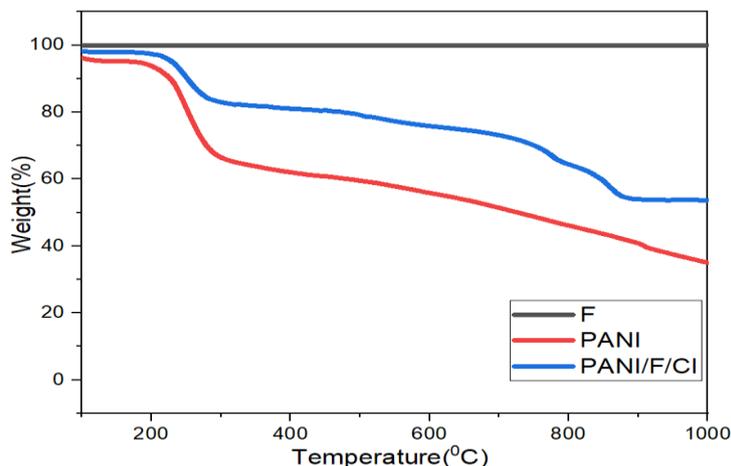


Figure 4. TGA thermograms of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , PANI/F/CI composite and pure PANI

### Morphology investigations

Figure 5 designates the morphology of the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite. The agglomerated spherical particles of NiZn ferrite and the spherical particles of carbonyl iron (Figure 5a, b) are observed with average diameters to be ranging between 27–63 nm and 0.2–2.4  $\mu\text{m}$ , respectively. While a combination of rough surface sheets and short rods connected to each other of PANI is noticed (Figure 5c), distributed in the range between 60–220 nm. On the other hand, after coating with polyaniline, a continued overlayer of PANI is created on the CI and  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles' surface (Figure 5d).

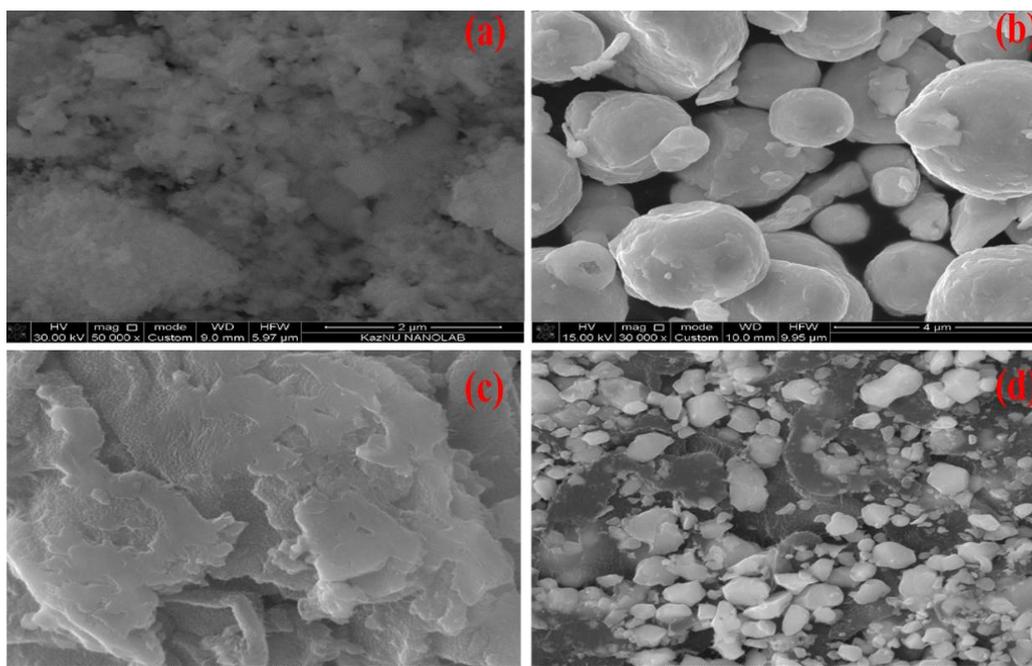


Figure 5. SEM images of (a)  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , (b) CI, (c) PANI and (d) PANI/F/CI composite

*Energy-dispersive X-ray spectroscopy (EDX) analysis*

Figure 6 and Table 1 present the energy-dispersive X-ray spectroscopy (EDX) analysis of  $\text{Ni}_{10.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , PANI and PANI/F/CI composite. The presence of C, O, Cl, S, Fe, Ni, Al, and Zn elements in the NiZn ferrite EDX spectrum is noticed. On the other hand, the presence of C, O, S and Cl elements in the PANI EDX spectrum is found. The presence of elemental Cl and S can be attributed to doping agents' hydrochloric acid and sodium dodecyl sulfate. Finally, the presence of C, O, Cl, S, Fe, Zn, Al, and Ni elements in the PANI/F/CI composite EDX spectrum is observed.

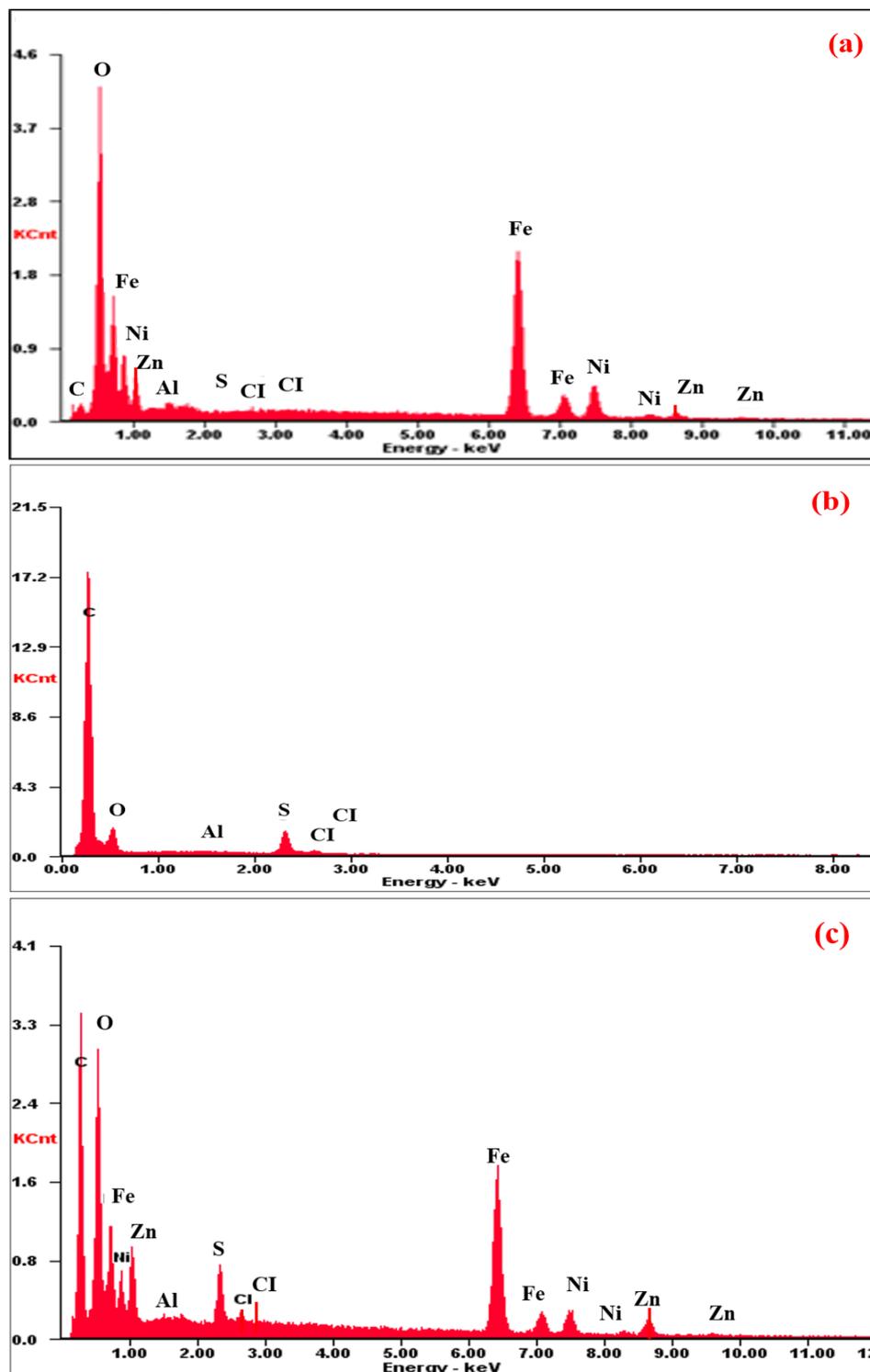


Figure 6. EDX of (a)  $\text{Ni}_{10.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , (b) PANI and (c) PANI/F/CI composite

EDX element composition of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , PANI and PANI/F/CI composite

Element	C	Cl	O	S	Al	Fe	Ni	Zn
Ferrite (wt %)	3.30	0.13	19.26	0.02	0.41	53.02	13.04	10.82
PANI (wt %)	84.52	0.45	11.81	3.17	0.05	0	0	0
PANI/F/CI (wt %)	51.72	0.51	19.23	2.02	0.08	17.15	5.32	3.97

*MA and EMI shielding properties*

MA and EMI shielding properties of the prepared samples are estimated with the free-space technique as illustrated in the following literature [27–31]. SE is calculated for the EMI shielding by applying the equation (1) [32]:

$$SE (dB) = SE_R + SE_A + SE_M = 10 \log \frac{P_{in}}{P_T}, \quad (1)$$

where  $p_{in}$  and  $p_T$  — the incident power and transmitted power of the EM waves, respectively.

It is significant to note that the multiple reflection loss ( $SE_M$ ) can be ignored if the absorption shielding ( $SE_A$ ) of EMI shielding material is higher than 10 dB and equation (1) then can be rewritten as [32]:

$$SE (dB) = SE_R + SE_A = 10 \log \frac{P_{in}}{P_T}. \quad (2)$$

The shielding by reflection ( $SE_R$ ) is calculated for the EMI shielding by applying the equation (3):

$$SE_R (dB) = -10 \log(1 - R) = -10 \log \left( 1 - \frac{P_{ref}}{P_{in}} \right). \quad (3)$$

The shielding by absorption ( $SE_A$ ) is calculated by equation (4) [33, 34]:

$$SE_A (dB) = -10 \log \left( \frac{T}{1 - R} \right) = -10 \log \left( \frac{P_T}{P_{in} - P_{ref}} \right), \quad (4)$$

where  $p_{ref}$  — the reflected power of the EM waves.

On the other hand, RL is calculated for the MA by applying the equation (5) [33, 34]:

$$Rl (dB) = 10 \log \frac{P_{in}}{P_{ref}}. \quad (5)$$

*Influence of the incorporation of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI and PANI on the RL and the SE*

EMI shielding and MA properties of the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite are studied. The results of this investigation are exhibited in Figures 7, 8 and Table 2. Figures 7, 8 illustrate the changing of the RL and SE as a function of the EM wave frequency for  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite. The absorption of samples with specified thickness at 3.2 mm is molded to measure RL and SE in the frequency band of 8.8–12.0 GHz. As illustrated in Figures 7, 8, a weak reflection loss and low shielding efficiency for the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  and CI are noticed. On the other hand, for the pure PANI, the reflection loss is in the range between 6.6–8.5 dB and the shielding efficiency is in the range between 9.8–12.9 dB. Furthermore, when PANI is incorporated with ferrite which is mixed with carbonyl iron, the reflection loss increases to –25.8 dB at 11.3 GHz for PANI/F/CI composite and the shielding efficiency increases to 30.12 dB at 11.0 GHz. Table 2 shows the reasonable surface density (SD) of all the prepared absorbers. As a result, one can notice the impact of incorporating  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  and CI (magnetic loss materials) and PANI (dielectric loss material) on the EMI and MA properties of the prepared absorber. This incorporation leads to an effective and low thickness absorber with a wide  $\text{BW}_{-10\text{dB}}$  [35]. Figure 9 shows the  $SE_R$  and  $SE_A$  of the  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite with a thickness of 3.2 mm at the frequency of 11.0 GHz.

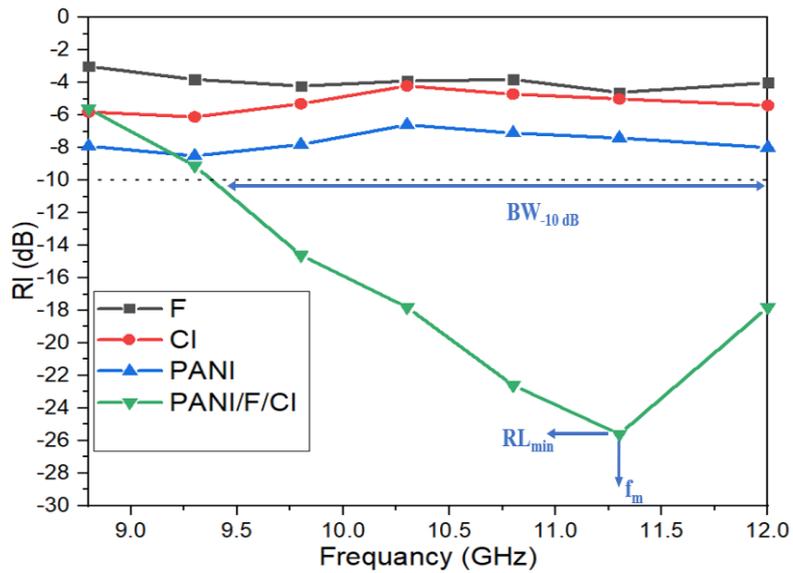


Figure 7. RL curves of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite at 3.2 mm thickness

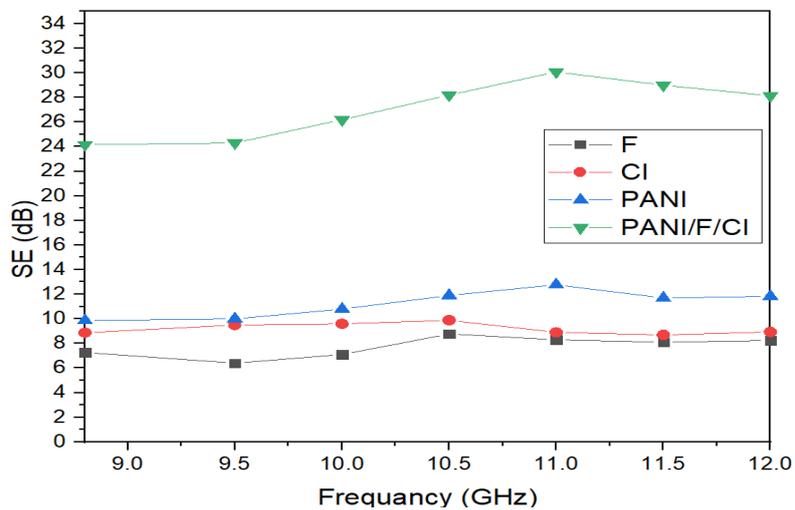


Figure 8. SE curves of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite at 3.2 mm thickness.

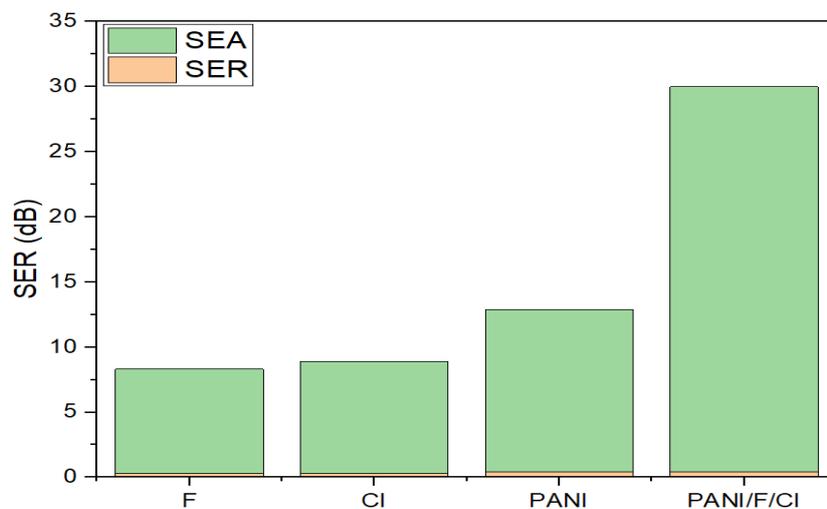


Figure 9. Bar plot for individual components of  $SE_R$  and  $SE_A$  of  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , CI, PANI and PANI/F/CI composite with a thickness of 3.2 mm at the frequency of 11.0 GHz

Table 2

MA behavior of Ni<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>, CI, PANI and PANI/F/CI composite at 3.2 mm thickness

Samples	RL <sub>min</sub> (dB)	f <sub>m</sub> (GHz)	BW <sub>-10dB</sub> (GHz)	SD (kg/m <sup>2</sup> )
Ni <sub>0.5</sub> Zn <sub>0.5</sub> Fe <sub>2</sub> O <sub>4</sub>	-4.6	-	-	4.56
CI	-6.5	-	-	5.21
PANI	-8.5	-	-	2.25
PANI/F/CI	-25.7	11.3	2.6	3.31

*Influence of the PANI/F/CI composite thickness and loading ratio on the RL*

Figure 10 illustrates the RL of PANI/F/CI composite with various thicknesses (3.2, 3.4, 3.6 mm) at the various weight ratios of the absorber within a paraffin matrix (30, 35 % w/w). It can be seen that the RL attenuation peaks of samples moved to lower frequencies with increasing sample thickness. This phenomenon may be defined by the quarter-wavelength ( $\lambda/4$ ) cancellation model, as shown in equation (6) [36–38]:

$$t_m = \frac{c}{4f_m \sqrt{|\mu_r||\epsilon_r|}}, \tag{6}$$

where  $|\epsilon_r|$  and  $|\mu_r|$  are the modulus of the measured complex relative permittivity ( $\epsilon_r$ ) and permeability ( $\mu_r$ ) at matching frequency ( $f_m$ ), respectively;  $c$  is the velocity of light.

It can be noticed from equation (6) that the  $f_m$  is inversely proportionate to the thickness of an absorber. On the other hand, one can notice the minimum reflection loss moves gradually to a lower frequency with the increase in weight ratios of the absorber within a paraffin matrix (Figure 10). Furthermore, Table 3 shows the PANI/F/CI composites have reasonable surface density, ranging from 3.31 to 3.40 kg/m<sup>2</sup>, and wide bandwidth extending from 2.5 to 3.0 GHz. One can conclude that optimal absorption can be accomplished by modifying the absorber thickness and the loading ratio of the absorber within a paraffin matrix.

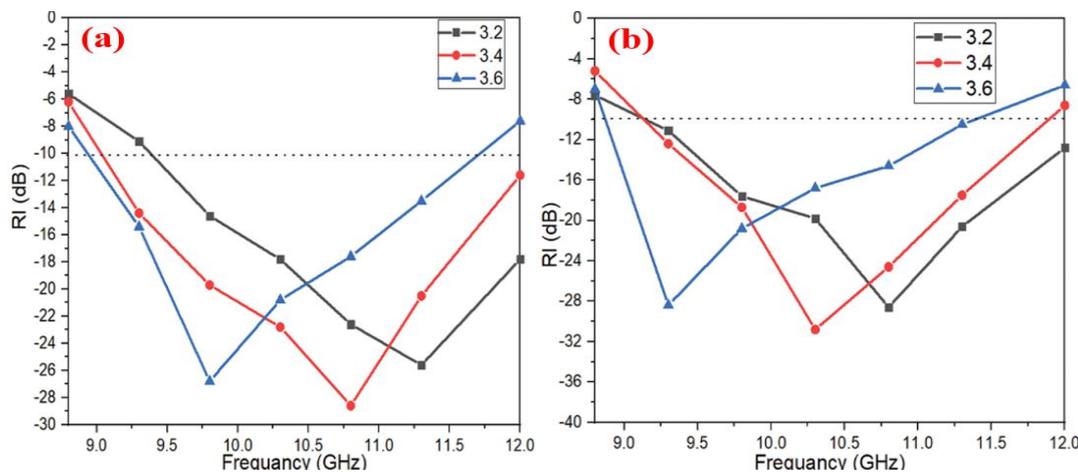


Figure 10. RL curves of PANI/F/CI composite with various thicknesses (3.2, 3.4, 3.6 mm) at the various weight ratios of the absorber within a paraffin matrix (a) 30 % and (b) 35 %

Table 3

MA behavior of PANI/F/CI composite at various thicknesses and various loading ratios within a paraffin matrix

Loading ratio %	t (mm)	RL <sub>min</sub> (dB)	f <sub>m</sub> (GHz)	BW <sub>-10dB</sub> (GHz)	SD (kg/m <sup>2</sup> )
30 %	3.2	-25.7	11.3	2.6	3.31
	3.4	-28.5	10.8	3.0	3.35
	3.6	-26.8	9.8	2.9	3.37
35 %	3.2	-28.6	10.8	2.5	3.33
	3.4	-30.8	10.3	2.8	3.38
	3.6	-28.4	9.3	2.6	3.40

To evaluate the beneficial impact of adding CI to the PANI/F on the microwave properties of the microwave absorber, Table 4 shows a comparison of MA properties of some lately reported PANI/NiZn ferrite absorbers with various loading ratios of the composites in the host matrix. Compared to PANI/NiZn ferrite absorbers reported by Ting et al. [3], Wang et al. and Wang et al. [7, 8], the current nanocomposite absorbers as-presented in this study display better MA in the frequency range of 8–12 GHz, lower loading ratio of the absorbers in the host matrix and relatively wider absorption bandwidth. However, the microwave absorber lately designed by Wang et al. [6] has comparable results with the current absorbers in terms of bandwidths and reflection losses, the lower loading ratios of the current absorbers are still an advantage. On the other hand, Didehban et al. designed absorbers with a low loading ratio similar to the ratio used in this study [5]. Although, the microwave properties of the current composites possess better reflection losses and larger bandwidths comparable with the result presented by Didehban et al. [5].

Table 4

Comparison of MA behavior of the current composites with similar absorbers in the literature

Specimen (relative weight ratio)/ host matrix	Loading percentage in the host matrix (%)	t (mm)	RL <sub>min</sub> (dB)	f <sub>m</sub> (GHz)	BW <sub>-10dB</sub> (GHz)
PANI/F/CI/ Paraffin (current work)	30	3.4	-28.5	10.8	3.0
	35	3.4	-30.8	10.3	2.8
NiZn ferrite-PANI(50:50)/Epoxy [3]	67	2.00	-14	11.0	2.6
NiZn ferrite-PANI(35:65)/Epoxy [5]	20	2.00	-20	9.1	0.5
NiZn ferrite-PANI(1:3)/Paraffin [6]	75	3.50	-32	9.5	3.8
NiZn ferrite-PANI(2:1)/Paraffin [7]	70	2.00	-27.5	6.0	3.0
NiZn ferrite-PANI/Epoxy [8]	67	3.00	-17	11.1	2.8

These results prove that adding CI to the PANI/F has a beneficial role in enhancing the microwave properties of the absorber: It becomes lighter with wide bandwidth.

### Conclusions

In the current research, we succeeded in the preparation of wideband and lightweight PANI/F/CI ferrite microwave absorbers with low loading ratios in paraffin wax 30–35 % w/w. The composites were structurally characterized using X-ray diffractometry, FTIR spectroscopy, UV-vis spectroscopy, and TGA. The morphology of the composites was investigated by scanning electron microscopy. The functional characterization was accomplished by measuring the EMI shielding and MA properties. The results show that by adequate control of the loading ratio and thickness of the absorber, one can tailor the design of a wideband and lightweight absorber based on PANI/F/CI in the frequency band of 8.8–12 GHz. Minimal reflection loss of -30.8 dB at the matching frequency of 10.3 GHz and the absorption bandwidth under -10 dB of 2.8 GHz for 3.4 mm thickness with a surface density of 3.38 kg/m<sup>2</sup> were noticed for the PANI/F/CI microwave absorber. The maximum shielding efficiency of 30.12 dB at 11.0 GHz for 3.2 mm thickness was observed for the PANI/F/CI microwave absorber.

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## Парафинді балауыз матрицасында полианилинмен қапталған Ni<sub>0,5</sub>Zn<sub>0,5</sub>Fe<sub>2</sub>O<sub>4</sub>/CI композитінің синтезі және микротолқынды сіңіру қасиеттері

Полианилин/Ni<sub>0,5</sub>Zn<sub>0,5</sub>Fe<sub>2</sub>O<sub>4</sub>/карбонил темірінің үштік композиттері (PANI/F/CI) екі кезең арқылы дайындалды: алдымен, Ni<sub>0,5</sub>Zn<sub>0,5</sub>Fe<sub>2</sub>O<sub>4</sub> золь-гель әдісімен; содан соң PANI/F/CI композиттері Ni<sub>0,5</sub>Zn<sub>0,5</sub>Fe<sub>2</sub>O<sub>4</sub> және CI болған кезде PANI-ның in-situ полимерлеу әдісі қолданылған. Үлгілерді сипаттау үшін рентгендік дифрактометрия (XRD — X-ray diffractometry), Фурье түрлендіру инфрақызыл спектроскопиясы (FTIR — Fourier transform infrared), ультракүлгін-көрінетін спектроскопиясы және термогравиметриялық талдау (TGA — Thermogravimetric analysis) пайдаланылды. Ұнтақтардың морфологиясы сканерлеуші электронды микроскоппен (SEM — Scanning electron microscope) зерттелген. Электромагниттік кедергілерді (EMI — electromagnetic interference) қорғау және микротолқынды жұту (MA — microwave absorption) қасиеттері 8,8–12 ГГц жиілік диапазонында өлшенді. Нәтижелер микротолқынды сіңіру қасиеттері абсорбердің қалыңдығына және парафиндік матрицадағы абсорбердің жүктеме қатынасына байланысты екенін көрсетті. Қалыңдығы 3,4 мм, бетінің тығыздығы 3,38 кг/м<sup>2</sup> болатын PANI/F/CI композиттік үлгісі үшін 10,3 ГГц сәйкес жиілікте ( $f_m$ ) ең аз кері жоғалуы –30,8 дБ және жұту жолағы үшін 2,8 ГГц кезінде –10 дБ-ден (BW<sub>-10dB</sub>) төмен болатыны анықталды. 3,2 мм қалыңдықтағы PANI/F/CI композиттік үлгісі үшін 11,0 ГГц жиілікте 30,12 дБ максималды экрандау тиімділігі байқалды.

*Кілт сөздер:* полианилин, карбонилді темір, композиттер, шағылысу жоғалуы, абсорбциялық жолақ ені.

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**Синтез и микроволновые поглощающие свойства композита  $\text{Ni}_{0,5}\text{Zn}_{0,5}\text{Fe}_2\text{O}_4/\text{CI}$ ,  
покрытого полианилином в парафиновой матрице**

Тройные композиты полианилин/ $\text{Ni}_{0,5}\text{Zn}_{0,5}\text{Fe}_2\text{O}_4$ /карбонильное железо (PANI/F/CI) получены двумя стадиями: сначала методом золь–гель был получен  $\text{Ni}_{0,5}\text{Zn}_{0,5}\text{Fe}_2\text{O}_4$ , после этого был получен композит PANI/F/CI с использованием технологии полимеризации PANI в присутствии  $\text{Ni}_{0,5}\text{Zn}_{0,5}\text{Fe}_2\text{O}_4$  и CI. Для характеристики образцов использовались рентгеновская дифрактометрия (XRD), инфракрасная спектроскопия с преобразованием Фурье (FTIR), спектроскопия в ультрафиолетовом и видимом (UV-vis) спектрах и термогравиметрический анализ (TGA). Морфологии порошков были исследованы с помощью сканирующего электронного микроскопа (СЭМ). Свойства экранирования электромагнитных помех и поглощения микроволн измерялись в полосе частот 8,8–12 ГГц для исследования микроволновых характеристик. Результаты относятся к тем свойствам поглощения микроволн, которые связаны с толщиной поглотителя и коэффициентом загрузки поглотителя в парафиновой матрице. Для композитного образца PANI/F/CI были определены, что на частоте согласования 10,3 ГГц ( $f_m$ ) минимальные потери на отражение составляют –30,8 дБ и полоса поглощения ниже –10 дБ (BW<sub>10дБ</sub>) на частоте 2,8 ГГц для толщины 3,4 мм с поверхностной плотностью 3,38 кг/м<sup>2</sup>. Для образца композита PANI/F/CI с толщиной 3,2 мм наблюдалась максимальная эффективность экранирования 30,12 дБ на частоте 11,0 ГГц.

*Ключевые слова:* полианилин, карбонильное железо, композиты, потери на отражение, ширина полосы поглощения.

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