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Synthesis and Microwave Absorption Properties of ($Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB) Ternary Composites

In this work, ternary composites of NiZn ferrite/carbonyl iron/carbon black ($Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB) were prepared via two stages. Firstly, $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was prepared by a self-combustion method using sucrose as a fuel. After that, the operation was continued via mixing CB, CI, and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ through grinding balls. Three various weight ratios of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB (1:1:1, 1:1:2, and 2:1:1) with various thicknesses (2–4–6 mm) were prepared. The absorbers were prepared by dispersing ($Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB) composites with a weight ratio within a paraffin wax matrix of 40 % w/w. X-ray diffractometry and FTIR spectroscopy were used in order to characterize the samples. The morphology of the powders was investigated by SEM. The functional characterization was accomplished by measuring the microwave absorption properties in the frequency band of 8.8–12 GHz. The microwave absorption materials (MAMs) showed wide bandwidths under –10 dB in the range of 2.81–3.20 GHz and reasonable surface density in the range of 3.625–4.041 kg/m^2. The absorber of 3.20 GHz bandwidth had a minimal reflection loss of –19.4 dB at the matching frequency of 9.92 GHz with a thickness of 6 mm.

Keywords: NiZn ferrite; Carbonyl iron, Carbon black, Reflection loss, Absorption bandwidth, Matching frequency, Paraffin wax matrix, Surface density.

Introduction

Electromagnetic interference (EMI) would be regarded as an unwanted result of modern technology that has dangerous effects on human health, intelligent devices, and military industries. Consequently, this EMI has become a critical worldwide issue and its alleviation could be accomplished only by utilize of EMI shielding materials. Nowadays, several magnetic loss materials such as hexagonal ferrites, spinel ferrites, and carbonyl iron or dielectric loss materials such as conductive polymers and carbonaceous materials have played a significant role in 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. In recent years, MA composites based on carbon, carbonyl iron, and ferrite, have obtained significant concern due to their excellent electrical and ferrimagnetic characteristics. Carbonaceous materials-based composites have pulled in major attention for microwave absorption lately because of the unique structure of carbon-based materials. Carbon black is usually used to fit the requirements of high-effective microwave attenuation materials because of its superior characteristics, for example, high permittivity, high specified surface region, huge interface, etc. Carbon black has a unique place in the band of elevated-frequency 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 [1, 2]. When NiZn ferrite and carbonyl iron are mixed with carbon black, the MA characteristics of the resultant composite are anticipated to enhance. According to this, $BaFe_{12}O_{19}$/CI absorbers with various powder ratio compositions in the frequency range of 2–18 GHz were successfully prepared by Feng et al. [3]. The single-layer and double-layer absorbers were prepared, and their MA characteristics were studied. The outcomes showed that the double-layer absorber was clearly more than that of the single-layer absorber. Where the reflection loss (RL) for the double-layer absorber was –13 dB in the frequency range (6–18 GHz) and less than –8 dB in the frequency range (2–18 GHz). The thicknesses of the absorbers were 3.6 and 3.7 mm, respectively. On the other hand, Yan et al. [4] prepared a

mixture of doped polyaniline (PANI) coated porous structure carbonyl iron powder (CIP) and graphene sheets. The results showed that the absorption bandwidth under –10 dB ($BW_{-10\,dB}$) was 4.6 GHz for 2 mm thickness for the composites with 40 wt.% of PANI@ porous CIP and 5 wt.% of Graphene. The outcomes showed that graphene-PANI@ porous CIP was a promising wave absorbing composite material. Until now, to the best of our knowledge, no studies have been reported on the MA properties of composites made up of NiZn ferrite/carbonyl iron/carbon black ($Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB). In this work, a perfect absorber was obtained by incorporating $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and CI (magnetic loss materials) and CB (dielectric loss material) within a paraffin wax matrix. Where we study the effect of different weight ratios of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB and its effect on the MA properties. A distinct feature of this work is that NiZn ferrite was synthesized through a self-combustion method utilizing polyvinyl alcohol (PVA) as a chelating agent. After that, the operation is continuous through mixing and grinding CB, CI, and NiZn ferrite by grinding balls.

Experimental

Chemicals: polyvinyl alcohol (PVA) (hydrolyzed, MW: 72000), nickel(II) nitrate hexahydrate ($Ni(NO_3)_2{\cdot}6H_2O$, 98.3 % purity), zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$, 98.7 % purity), and iron(III) nitrate nonahydrate ($Fe(NO_3)_3{\cdot}9H_2O$, 98.2 % purity) were purchased from TRADING COMPANY ANT, Russia. On the other hand, carbonyl iron (CI, 99.6 % purity)) and carbon black (CB, 99.5 % purity)) were purchased from Cabot Norit Company, Netherland.

Instruments used: A powder X-ray diffractometer (XRD, Rigaku Miniflex 600, Cu-Ka) was used for determining the crystal structures of the powders. Fourier Transform IR (FTIR) spectra were recorded on a Perkin Elmer spectrum 65 FTIR spectrometer in the range of 400–4000 cm^{-1}. A scanning electron microscope (FEI Quanta 200 3D) was used for determining the powders morphology. The microwave absorption properties of the prepared samples were calculated by using the horn antenna connected to an oscilloscope (AKTAKOM ADS-2221M).

Methodology:

1. Synthesis of NiZn ferrite, carbonyl iron and carbon black powders

Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) nanoparticles were prepared by a self-combustion method. $Ni_{0.5}Zn_{0.5}Fe_2O_4$ were synthesized by taking appropriate amounts of ($Ni(NO_3)_2{\cdot}6H_2O$), ($Zn(NO_3)_2{\cdot}6H_2O$), and ($Fe(NO_3)_3{\cdot}9H_2O$). They were blended together with an aqueous solution of sucrose (2 moles per metal ion) and 1 % an aqueous solution of polyvinyl alcohol. The whole mixture was blended totally and heated at 90 °C for 7 h to shape a viscous liquid. The heating process was accompanied by the evolution of brown fumes of NO_2 from the decomposed metal nitrate salts. Then, the mixture was transferred to the furnace for drying for 2 h at 200 °C to obtain a fluffy carbonaceous pyrolyzed mass. After that, the resulting mass was annealed for 4 h at 650 °C to obtain nanoparticles of ferrite. Typical images of a prepared ferrite by a self-combustion method are shown in Figure 1. On the other hand, CI and CB were purchased from Cabot Corporation Company. The average particle size of CI and CB powders was measured utilizing the sieve shaker and it was between 10–25 μm and 2–8 μm, respectively. CI and CB powders were milled for 12 h at 300 rpm via grinding balls to obtain fine powders.

2. Synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB composites

Ferrite nanoparticles were mixed and milled with CI and CB powders by grinding balls. Three various weight ratios of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB (1:1:1, 1:1:2, and 2:1:1) were synthesized. The $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB composites were milled for 1 h at 300 rpm.

3. Preparation of absorber samples

Paraffin wax was symmetrically blended with $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB composites powders with a weight ratio of 40 % w/w within a paraffin wax matrix by heating and stirring for 15 min. Afterward, the single-layer samples were molded to the dimensions of 50×50 mm with various thicknesses (2–4–6 mm) to measure RL in the frequency band of 8.8–12 GHz.

4. Experimental setup for measuring reflection loss (RL)

The metal plate is put to reflect the transmitted power of the EM waves as shown in Figure 2. As a consequence, the transmitted power of the EM waves (p_t) is negligible in microwave absorption. Microwave absorption properties of the prepared samples are estimated with the free-space technique as shown in Figure 3. EM waves are generated by a microwave generator in the frequency band of 8.8–12 GHz, where a microwave generator is connected by a WR90 waveguide instrument (IEC Standard R100, X Band). The incident EM waves (p_{in}) are measured by the horn antenna connected to an oscilloscope (Fig. 3), then a prepared

sample is placed on the metal plate at an angle of 45° to measure the reflected power of the EM waves (p_{ref}) by an oscilloscope (Fig. 4). As a result, one can calculate the RL by applying the equation (1) [5, 6]:

$$Rl\left(dB\right)=10\log\frac{p_{in}}{p_{ref}}\,. \tag{1}$$

a — the metal salts aqueous solution in the flask; *b* — blending totally the whole mixture;
c — heating the solution at 90 °C; *d* — formation of a viscous liquid;
e — formation of a fluffy carbonaceous pyrolyzed mass; *f* — obtaining nanoparticles ferrite

Figure 1. Synthesis of NiZn ferrite nanoparticles by a self-combustion method

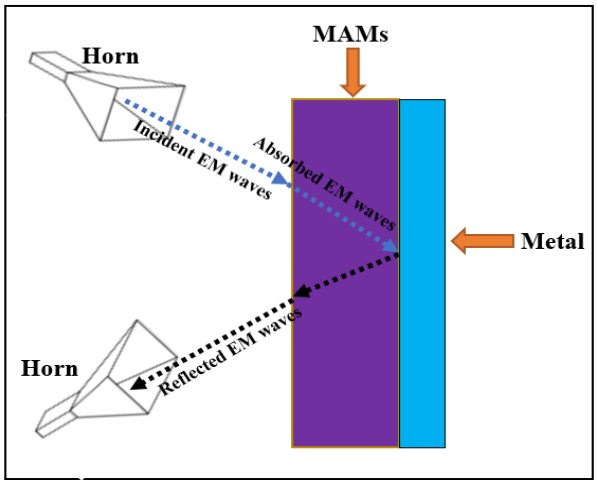
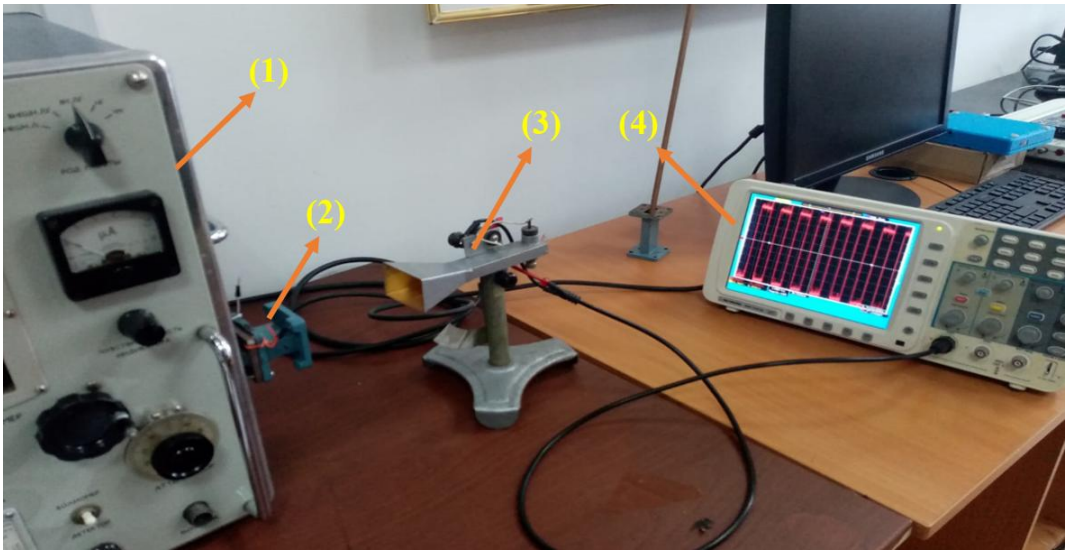


Figure 2. Sketch of the microwave absorption model used to measure reflection loss



1 — microwave generator; *2* — waveguide instrument (IEC Standard R100, X Band);
3 — horn antenna; 4 — oscilloscope

Figure 3. Experimental setup for measuring the incident power of the EM waves by the free-space technique

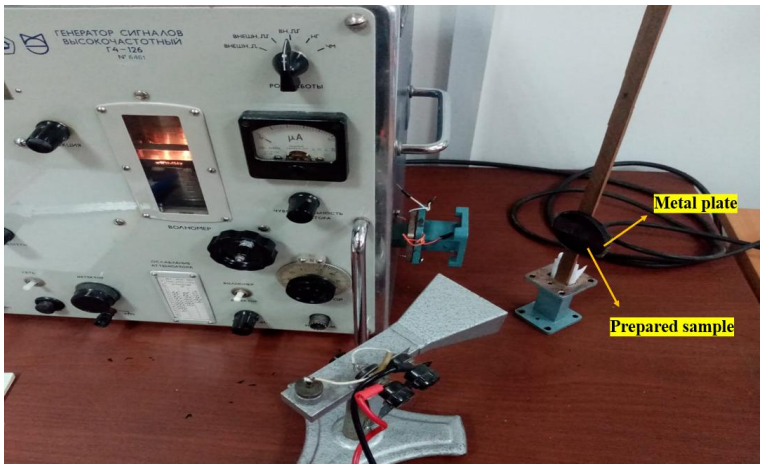


Figure 4. Experimental setup for measuring the reflected power of the EM waves by the free-space technique

5. Statistical processing of experimental data

Four samples of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB composites were designed for each thickness (2–4–6 mm) to measure the microwave absorption properties. Statistical processing is used to describe the experimental data. For an estimated statistic, we can use a confidence interval. The purpose of a confidence interval is to supplement the value estimate of the sample within the formation of the uncertainty in this estimate. The method estimate ± margin of error is used to get an interval based on each sample. The margin of error is given by the equation (2) [7]:

$$\text{Margin of error} = Z^* \cdot \frac{\text{standered deviation}}{\sqrt{n}}, \quad (2)$$

where Z^* — is the confidence level which is selected at 95 %. The value of Z^* for a specific confidence level is 1.96; n — is the size of the sample that is used to compute the margin of error.

The confidence interval is given by the equation (3) [7]:

$$\text{Confidence interval} = \text{Sample mean} \pm Z^* \cdot \frac{\text{standered deviation}}{\sqrt{n}}. \quad (3)$$

Results and discussion

XRD patterns

The XRD patterns of $Ni_{0.5}Zn_{0.5}Fe_2O_4$, CI and CB powders are shown in Figure 5. For the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ pattern, nine diffraction peaks were observed, which conformed to (hkl) planes of (111), (220), (311), (222), (400), (422), (511), (440) and (533), respectively. The ideal spinel structure was observed by the peaks of NiZn ferrite [8]. All the observed peaks of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ matched with the standard XRD pattern (JCPDS, PDF no. 08–0234). On the other hand, for the carbonyl iron pattern, three characteristic peaks were observed, which conformed to (hkl) planes of (100), (200), and (211), respectively. The XRD pattern of carbonyl iron resembles crystallites in which the sample mainly contains α-Fe phase [9]. All the observed peaks of CI matched with the standard XRD pattern (JCPDS, PDF no. 06-0696). Finally, for the CB pattern, two characteristic peaks were observed, which conformed to (hkl) planes of (002) and (100), respectively [10].

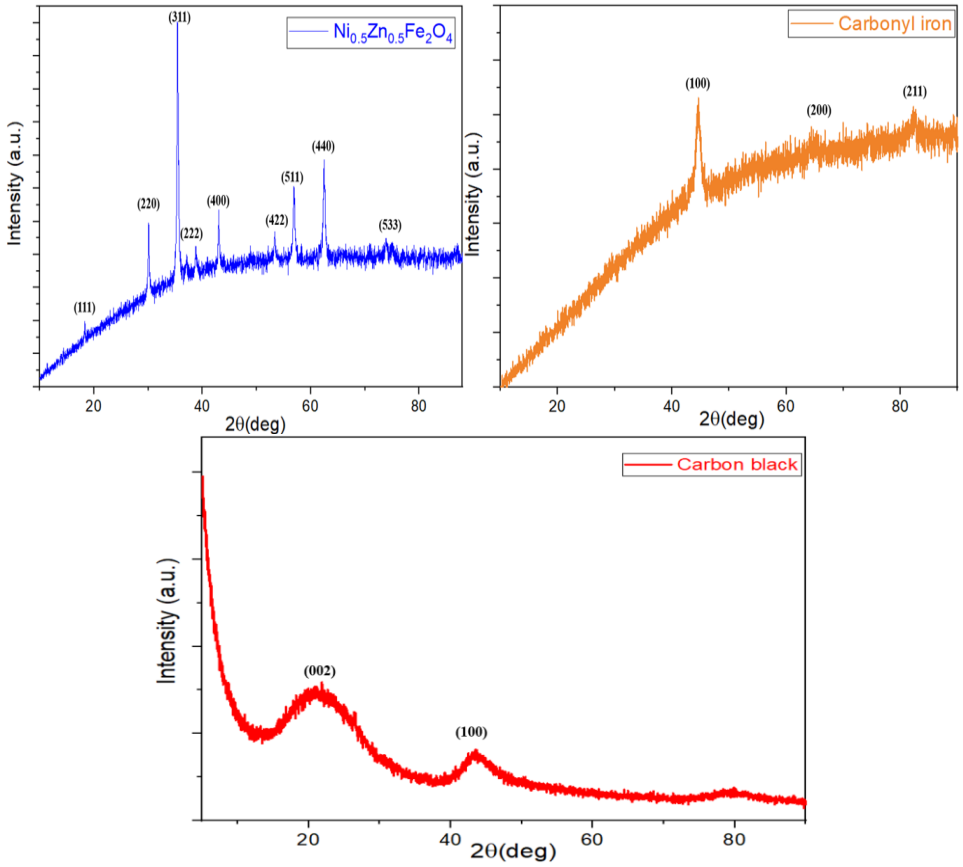


Figure 5. XRD patterns of $Ni_{0.5}Zn_{0.5}Fe_2O_4$, carbonyl iron and carbon black

FTIR spectra

The FTIR spectrum of the $Ni_{0.5}Zn_{0.5}Fe_2O_4$, CI and CB powders is shown in Figure 6. For the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles, two peaks at 565.4 cm^{-1} and 432.3 cm^{-1} are referred to the stretching vibration of (Fe-O), which emphasizes the forming of the metal-oxygen in ferrite-based [11]. On the other hand, the peak at 1630.4 cm^{-1} in $Ni_{0.5}Zn_{0.5}Fe_2O_4$, CI, and CB is referred to C=O stretching vibration, and the peaks at 2348 cm^{-1} and 3452 cm^{-1} are referred to O-H stretching vibration [12, 13].

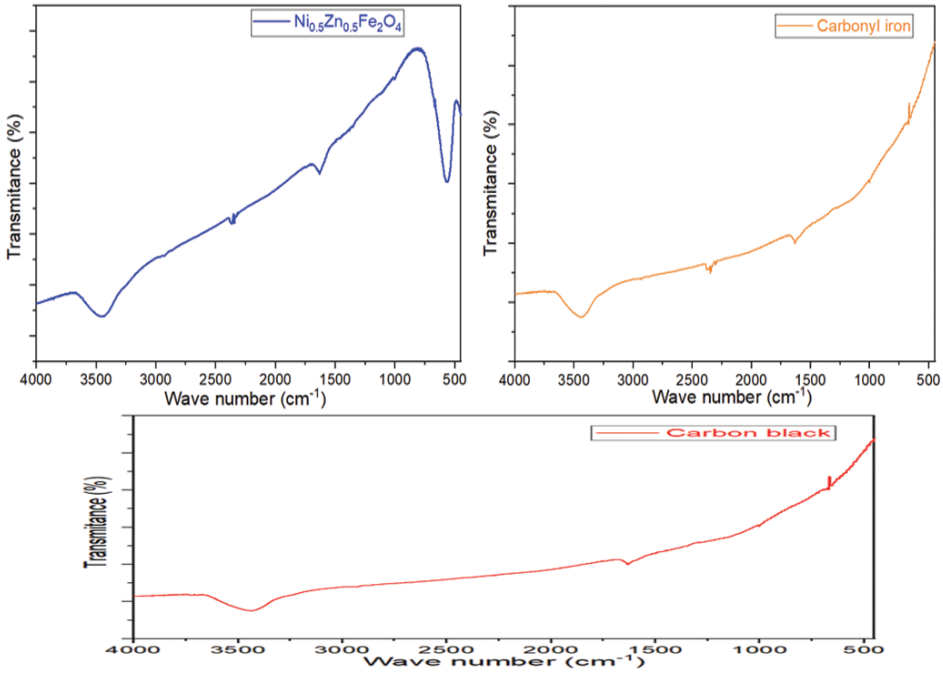
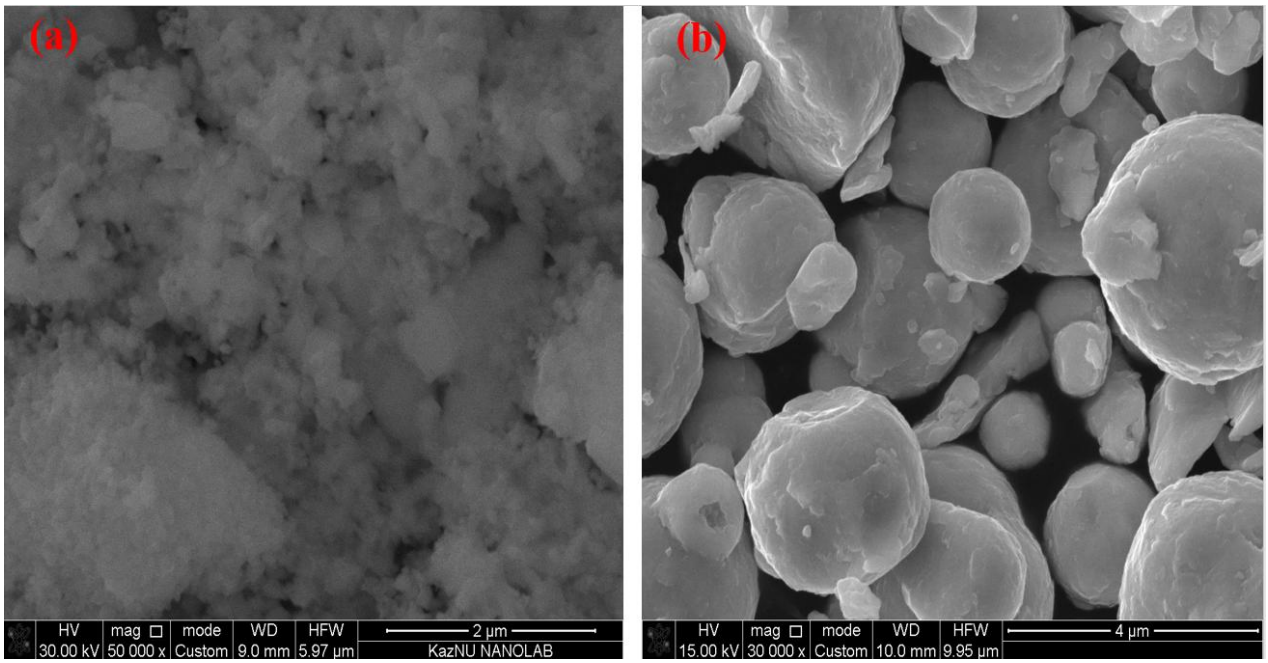


Figure 6. FTIR spectra of NiZn ferrite, carbonyl iron and carbon black

SEM analysis

The powders morphology was investigated by SEM. The agglomerated spherical particles of NiZn ferrite and the spherical particles of carbonyl iron (Fig. 7*a*, *b*) were observed with the average diameters to be ranging between 18–52 nm and 0.2–2.4 μm, respectively. On the other hand, the average particle size of carbon black powder (Fig. 7*c*) was found to be ranging between 75–481 nm.



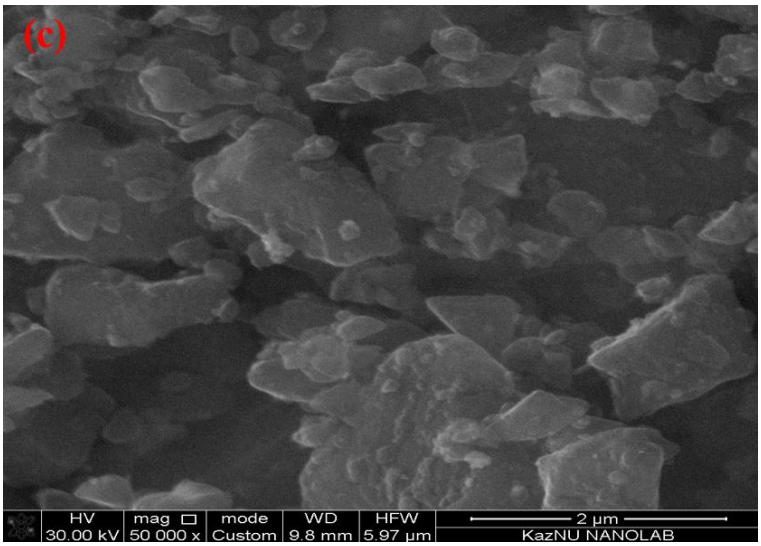


Figure 7. SEM images of (a) NiZn ferrite, (b) carbonyl iron and (c) carbon black

Microwave absorption properties

MA properties of the F/CI/CB composites with various thicknesses (2–4–6 mm) at the weight percentage of the absorber within a paraffin matrix (40 % w/w) were studied. The results of this investigation are exhibited in Figure 8 which illustrates the changing of the RL as a function of the EM wave frequency. The RL of the prepared samples was measured by the variation between the power of incident (p_{in}) and reflected (p_{ref}) EM waves as shown in Figure 3 and Figure 4. The RL curves of the samples were obtained from equation (1). The $BW_{-10\,dB}$, RL_{min} and f_m were deduced from RL curves. Figure 8 shows that the RL attenuation peaks of samples moved to lower frequencies with increasing sample thickness. This phenomenon may be defined by the quarter-wavelength (λ/4) cancellation model, as shown in equation (4) [14–16]:

$$t_m = \frac{c}{4tf_m\sqrt{|\mu_r||\varepsilon_r|}}, \tag{4}$$

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

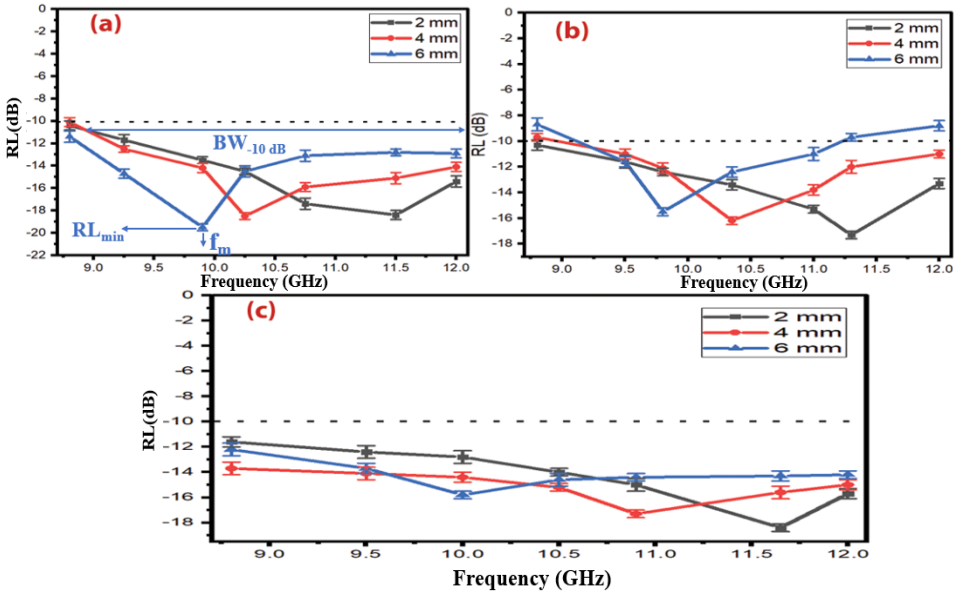


Figure 8. RL curves of (*a*) F/CI/CB-111 composite; (*b*) F/CI/CB-112 composite and (*c*) F/CI/CB-211 composite at various thicknesses (2–4–6 mm)

It can be noticed from equation (4) that the f_m is inversely proportionate to the thickness of an absorber. Furthermore, Table 1 shows the F/CI/CB composites have reasonable surface density, ranging from 3.625 to 4.041 kg/m^2, and wide bandwidth extending from 2.81 to 3.20 GHz. The absorber of 3.20 GHz bandwidth has a minimal reflection loss of –19.42 dB at the matching frequency of 9.92 GHz with a thickness of 6 mm. One can conclude that the optimal absorption can be accomplished by modifying the absorber thickness of the absorber within a paraffin matrix. In addition to that, one can notice the impact of incorporating $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and CI (magnetic loss materials) and CB (dielectric loss material) on the MA properties of the prepared absorber. This incorporation leads to an effective and low thickness absorber with a wide bandwidth under –10 dB [17]. Statistical processing was used to describe the experimental data, as the experiments showed the effect of sample density on microwave absorption properties (Table 1), as a result, the confidence interval on the microwave absorption properties was studied.

Table 1

MA behavior of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CI/CB composites at various thicknesses (2–4–6 mm)

Composite samples	*t* (mm)	RL_{min} (dB)	f_m (GHz)	$BW_{-10\,dB}$ (GHz)	*SD* (kg/m^2)
F/CI/CB-111	2	–18.32±0.86	11.52±0.25	3.20±0.01	3.861±0.007
	4	–18.54±0.71	10.34±0.36	3.20±0.01	3.874±0.006
	6	–19.42±0.83	9.92±0.14	3.20±0.01	3.892±0.004
F/CI/CB-112	2	–17.36±0.62	11.34±0.37	3.20±0.01	3.625±0.005
	4	–16.33±0.86	10.41±0.16	2.92±0.05	3.643±0.005
	6	–15.58±0.73	9.82±0.23	2.81±0.03	3.652±0.008
F/CI/CB-211	2	–18.46±0.71	11.73±0.13	3.20±0.00	4.012±0.009
	4	–17.31±0.79	10.96±0.38	3.20±0.00	4.027±0.007
	6	–15.82±0.61	10.00±0.35	3.20±0.00	4.041±0.006

Conclusion

F/CI/CB microwave absorbers were synthesized within a paraffin matrix successfully. $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and CI were used to enhance the mechanism of magnetic loss, while CB was introduced to enhance the mechanism of dielectric loss. As a result, one can notice the impact of combining $Ni_{0.5}Zn_{0.5}Fe_2O_4$, CI and CB on the MA properties of the absorber. This combination leads to an effective and low thickness absorber with a wide BW_{-10dB}. The results refer that by sufficient control of the thickness of the absorption material and the weight ratio of F/CI/CB, we can design a wideband absorber based on F/CI/CB in the frequency band of 8.8–12 GHz.

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Үштік композиттердің синтезі және микротолқынды сіңіру қасиеттері ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB)

Бұл жұмыста феррит NiZn/темір карбонил/ қара көміртек ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB) үштік композиттері екі кезеңде алынды: біріншіден, $Ni_{0,5}Zn_{0,5}Fe_2O_4$ өздігінен жану арқылы алынды. Одан кейін ұнтақтау шарлары арқылы CB, CI және $Ni_{0,5}Zn_{0,5}Fe_2O_4$ араластыруымен операция жалғасты. Әр түрлі қалыңдықтағы (2–4–6 мм) $Ni_{0,5}Zn_{0,5}Fe_2O_4$ (1:1:1, 1:1:2 және 2:1:1) үш түрлі салмақ қатынасы дайындалды. Абсорберлер салмағы бойынша 40 % парафинді балауыз матрицасында салмақ қатынасы бар композиттерді ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB) дисперсиялау арқылы жасалды. Үлгілерді сипаттау үшін рентгендік дифрактометрия және FTIR спектроскопиясы қолданылады. Ұнтақтардың морфологиясы SEM арқылы зерттелді. Функционалдық сипаттама 8,8–12 ГГц жиілік диапазонында микротолқынды сіңіру қасиеттерін өлшеу арқылы орындалды. Микротолқынды сіңіру материалдары 2,81–3,20 ГГц диапазонында –10 дБ-ден төмен кең өткізу қабілеттілігін және 3,625–4,041 кг/м^2 диапазонында қолайлы бет тығыздығын көрсетті. Өткізу жолағы 3,20 ГГц, қалыңдығы 6 мм болатын абсорбер 9,92 ГГц сәйкес жиілікте –19,4 дБ шағылудың минималды жоғалуына ие.

Кілт сөздер: NiZn ферриті, карбонилді темір, көміртегі қарасы, шағылысу жоғалтуы, сіңіру жолағы, сәйкестік жиілігі, парафинді балауыз матрицасы, бетінің тығыздығы.

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Синтез и микроволновые поглощающие свойства тройных композитов ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB)

В настоящей работе были получены тройные композиты феррит NiZn/карбонил железа/сажа ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB) в два этапа. Сначала методом самовозгорания был получен $Ni_{0,5}Zn_{0,5}Fe_2O_4$. После этого операция продолжалась путем перемешивания CB, CI и $Ni_{0,5}Zn_{0,5}Fe_2O_4$ через мелющие шары. Были изготовлены три различных весовых соотношения $Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB (1:1:1, 1:1:2 и 2:1:1) различной толщины (2–4–6 мм). Поглотители изготовляли путем диспергирования композитов ($Ni_{0,5}Zn_{0,5}Fe_2O_4$/CI/CB) с массовым соотношением в матрице парафинового воска 40 % по массе. Рент-

геновская дифрактометрия и FTIR-спектроскопия использовались для характеристики образцов. Морфология порошков была исследована с помощью СЭМ. Функциональная характеристика получена путем измерения характеристик поглощения микроволн в диапазоне частот 8,8–12 ГГц. Материалы, поглощающие микроволновое излучение (ПМИ), показывали широкую полосу пропускания ниже –10 дБ в диапазоне 2,81–3,20 ГГц и приемлемую поверхностную плотность в пределе 3,625–4,041 кг/м^2. Поглотитель с полосой пропускания 3,20 ГГц имеет минимальные потери на отражение –19,4 дБ на частоте согласования 9,92 ГГц при толщине 6 мм.

Ключевые слова: феррит NiZn, карбонильное железо, сажа, потери на отражение, ширина полосы поглощения, частота согласования, парафиновая матрица, поверхностная плотность.

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