

Research Article

Facile Preparation, Characterization, and Highly Effective Microwave Absorption Performance of CNTs/Fe₃O₄/PANI Nanocomposites

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A facile method has been developed to synthesize light-weight CNTs/Fe₃O₄/PANI nanocomposites. The formation route was proposed as the coprecipitation of Fe²⁺ and Fe³⁺ and an additional process of in situ polymerization of aniline monomer. The structure and morphology of CNTs/Fe₃O₄/PANI were characterized by transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and Fourier transform infrared (FTIR) spectroscopy. The TEM investigation shows that the CNTs/Fe₃O₄/PANI nanocomposites exhibit less intertwined structure and that many more Fe₃O₄ particles are attached homogeneously on the surface of CNTs, indicating that PANI can indeed help CNTs to disperse in isolated form. The wave-absorbing properties were investigated in a frequency of 2–18 GHz. The results show that the CNTs/Fe₃O₄/PANI nanocomposites exhibit a super absorbing behavior and possess a maximum reflection loss of –48 dB at 12.9 GHz, and the bandwidth below –20 dB is more than 5 GHz. More importantly, the absorption peak frequency ranges of the CNTs/Fe₃O₄/PANI composites can be tuned easily by changing the wax weight ratio and thickness of CNTs/Fe₃O₄/PANI paraffin wax matrix.

1. Introduction

With the rapid increase in the use of telecommunications, digital systems, and fast processors, electromagnetic interference (EMI) has become a great concern. A microwave absorber is a kind of functional material that can absorb electromagnetic (EM) waves effectively and convert EM energy into thermal energy or make EM waves dissipate by interference [1]. Over the past decade, a variety of materials used as microwave absorbers have been extensively studied with increasing demand for innovative EMI shielding [2–6]. Among the pursued materials, carbon nanotubes (CNTs) have attracted extensive development effort for their unique structural, high electrical conductivity, outstanding chemical stability, and mechanical properties [7–9]. However, excellent microwave absorption properties cannot be obtained for

unmodified CNTs because their magnetic loss is so small [10]. To optimize the performances of CNTs as microwave absorbers, it is necessary to modify CNTs by decorating other nanomaterials, which are expected to exhibit ideal electromagnetic absorption properties [11, 12].

In recent years, more and more research has been conducted in decorating CNTs with iron oxide and preparing magnetic nanoparticles due to their unique magnetic features, low cost, and strong absorption characteristics [13, 14]. Various chemistry-based processing routes have been developed to synthesize iron oxide/CNTs nanocomposite. Correa-Duarte et al. [15] coated CNTs with iron oxide nanoparticles (magnetite/maghemite) via a layer by layer assembly technique and aligned CNTs chains in relatively small external magnetic fields. Youn et al. [16] decorated

single walled carbon nanotubes (SWNTs) with iron oxide nanoparticles along the nanotube via a magnetoevaporation method. In addition, Jia et al. [17] initiated the self-assembly of magnetite particles along multiwalled CNTs via a hydrothermal process. However, there are still disadvantages in most of these processes, which mainly include the points as follows: (1) Fe_3O_4 materials usually suffer from ease of oxidation and relatively narrow absorption frequency range, which hamper their applications, and (2) the dispersion of the nanocomposite is poor, and the orientation of the deposition of Fe_3O_4 nanoparticles on the surfaces of CNTs is difficult to control.

In order to solve these problems, here in this report the CNTs/ Fe_3O_4 /PANI nanocomposites were fabricated by the coprecipitation of Fe^{2+} and Fe^{3+} and an additional process of in situ polymerization of aniline monomer. Polyaniline (PANI), polymerized from the inexpensive aniline monomer, was considered as an ideal matrix or a second phase incorporated with CNTs for its unique electromagnetic shielding and attenuation [18]. Furthermore, the facile preparation, structure characterization, and wave absorption performance of CNTs/ Fe_3O_4 /PANI nanocomposite materials were investigated. The effects of several factors on wave absorption properties of CNTs/ Fe_3O_4 /PANI nanocomposites were discussed.

2. Experimental Section

2.1. The Pretreatment of MWCNTs. MWCNTs (purity >95 wt%, 20–30 nm outer diameter, 10–30 μm length) were purchased from Chengdu Organic Chemicals Co. Ltd, Chinese Academy of Science. The as-supplied MWCNTs were processed in concentrated sulfuric acid with vigorous ultrasonic vibration treatment (30 W, 40 kHz) for 2 h at 50°C. After that, the mixture was followed by vacuum filtration, and washed with deionized water until the pH value of washing solution is neutral. Then, the filtered solid was dried under vacuum for 24 h at 60°C, obtaining acidulated MWCNTs.

2.2. The Preparation of CNTs/ Fe_3O_4 /PANI Nanocomposite Materials. $\text{NH}_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was employed as the precursor for the synthesis of Fe_3O_4 magnetic nanoparticles. $\text{NH}_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (1.0 g) was dissolved first in a mixture of deionized water (15 mL) and hydrazine hydrate solution (5 mL) with a volume ratio of 3:1 under constant stirring. Then, 0.4 g of acidulated MWCNTs was added into the above solution. After 30 min ultrasonic vibration, a black suspension with homogeneously dispersed MWCNTs was obtained. Ammonia water (1.0 mol/L) was then added until the pH value of the solution was 11.0. The stable aqueous suspension was subsequently long-drawn reflux condensed in a thermostatic water bath at the temperature of 100°C. After approximately 2 h, the products were cooled to room temperature. Meanwhile, the obtained products were quickly redispersed in 30 mL deionized water. Benzenesulfonic acid with certain concentration (1.5 mol/L) and aniline (0.2 g) was subsequently added. Until homogenous suspension was achieved, ammonium persulfate (APS) aqueous solution

(0.2 g of APS in 20 mL of deionized water) was dropwise added to the suspension. The polymerization process was applied in an ice bath for 6 h under vigorous stirring. The resulting precipitations were washed with deionized water and absolute ethanol several times to remove the remaining impurities. In the next step, the as-prepared product was dried under vacuum for 24 h at 60°C.

3. Characterization

The morphology and size distribution of the samples were investigated by means of transmission electron microscopy (TEM, JEM-2010F, Germany). The ferroferric oxide nanoparticle in CNTs/ Fe_3O_4 /PANI nanocomposites was examined specially by X-ray photoelectron spectroscopy (XPS). Fourier transform infrared spectroscopy (FTIR) was carried out on a Nicolet 8700 FTIR system. The reflection loss was measured with an Anritsu 37269D vector network analyzer in the 2–18 GHz range for which the samples were prepared by uniformly mixing the CNTs/ Fe_3O_4 /PANI nanocomposites in a paraffin matrix which is pressed in a cylindrical shape with thickness of 2.00 mm.

4. Results and Discussion

4.1. TEM Images of CNTs/ Fe_3O_4 /PANI and CNTs/ Fe_3O_4 . The typical morphology of the as-prepared CNTs/ Fe_3O_4 /PANI is shown in Figure 1(a). As a comparison, CNTs/ Fe_3O_4 samples prepared without PANI are presented in Figure 1(b), demonstrating smaller average diameter observed in the latter because of the nonexistence of PANI coating. What is more, in Figure 1(b) samples of CNTs/ Fe_3O_4 prepared without PANI seem to be a bundle that CNTs are intertwined with each other and that Fe_3O_4 magnetic nanoparticles aggregate closely. However, the samples prepared in the presence of PANI (shown in Figure 1(a)) exhibit less entangled structure due to the wrapping of PANI. In other words, the use of PANI as a dispersant in the preparation process of CNTs/ Fe_3O_4 can indeed help CNTs to disperse as individuals or smaller bundles. On the other hand, the TEM image of CNTs/ Fe_3O_4 /PANI also indicates that a large number of Fe_3O_4 nanoparticles adhered on the CNTs are distributed relatively homogeneously and with tiny diameters in the range of 3–5 nm; promising CNTs/ Fe_3O_4 /PANI nanocomposites prepared with PANI may have higher magnetic properties and absorption characteristics. Furthermore, it is noticeable that the interaction between Fe_3O_4 nanoparticles and CNTs was strong in the presence of PANI, as few Fe_3O_4 nanoparticles were observed on the copper grids suffering from repeated washing and sonicating in the process.

4.2. XPS Analysis of CNTs/ Fe_3O_4 /PANI. XPS has often been used for the surface characterization of various materials, and unambiguous results are readily obtained when the various surface components each contain unique elemental markers. Here, in order to further analyse the CNTs/ Fe_3O_4 /PANI products, XPS was measured to understand the composition of the magnetic nanoparticles surface in Figure 2. Figure 2(a)

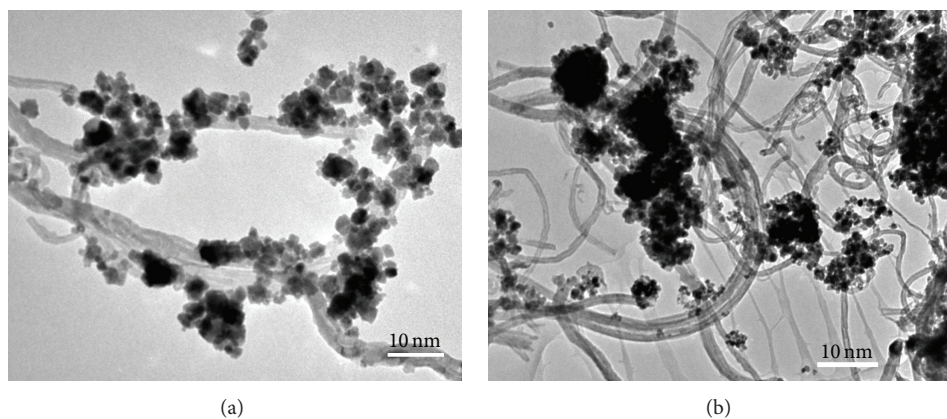


FIGURE 1: (a) TEM image of the as-prepared CNTs/Fe₃O₄/PANI; (b) TEM image of CNTs/Fe₃O₄.

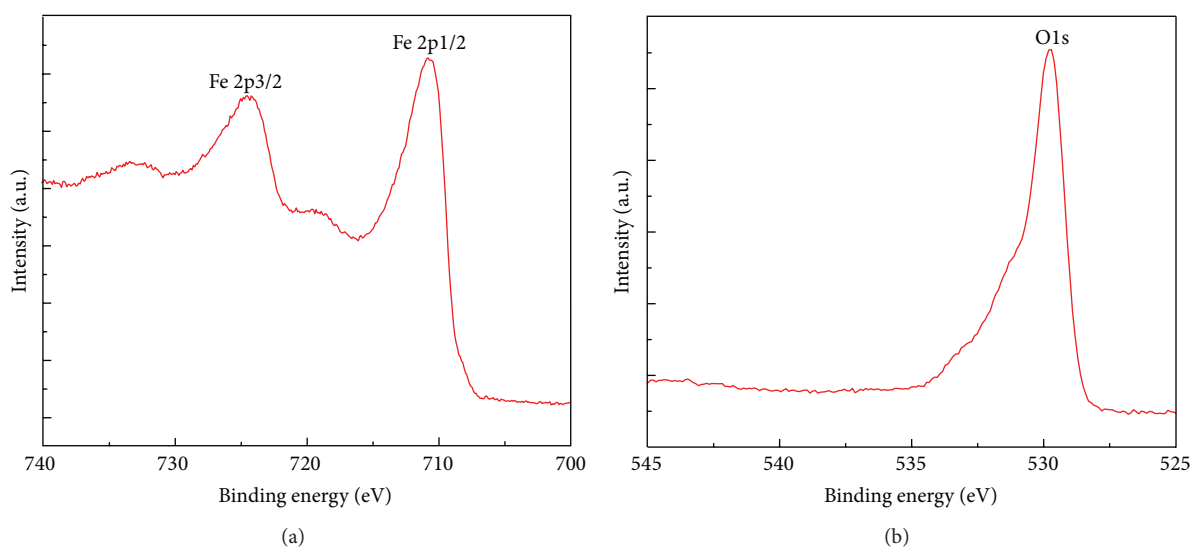


FIGURE 2: (a) XPS signal from Fe in Fe₃O₄ magnetic nanoparticles; (b) XPS signal from oxygen in Fe₃O₄ magnetic nanoparticles.

shows the XPS signals of the Fe 2p region. Two peaks of Fe 2p_{1/2} at 710.6 eV and Fe 2p_{3/2} at 723.9 eV were observed, respectively. The O 1s spectrum in Figure 2(b) consisted of a main peak originating from the oxygen in Fe₃O₄ (at 529.7 eV) and a shoulder centered at 531.3 eV, which has been ascribed to surface traps [19]. The Fe/O ratio was estimated as 0.74 with curve resolution analysis, which matches well with the stoichiometric ratio of Fe₃O₄ (0.75). The data are also consistent with the values reported for Fe₃O₄ in the literature [20–22]. Therefore, XPS results also prove the composition of the products.

4.3. FTIR Analysis of CNTs/Fe₃O₄/PANI. FTIR spectra of samples were recorded at room temperature, which are shown in Figure 3. Figure 3(a) shows the results of neat PANI. Figure 3(b) shows the CNTs/Fe₃O₄/PANI samples prepared in the presence of PANI. For the CNTs/Fe₃O₄/PANI nanocomposite, the peaks appearing at 3268, 1587, 1496, 1310, 1165, and 832 cm⁻¹ indicate the formation of PANI. The peaks at 1587 and 1496 cm⁻¹ are assigned to the stretching vibration

of C=N in quinoid ring and C=C in benzenoid ring [23]. The peak at 3268 cm⁻¹ is attributed to the N–H stretching vibration in PANI [23]. However, compared with the spectra of the PANI, the peaks of the CNTs/Fe₃O₄/PANI nanocomposites shifted slightly in the direction of low wavenumber. This may be attributed to the interaction between the CNTs and the PANI [24]. What is more, owing to lower concentrations of PANI in CNTs/Fe₃O₄/PANI samples and the introduction of the CNTs, the intensity of the peaks of the CNTs/Fe₃O₄/PANI nanocomposite decreased.

4.4. Reaction Mechanism Explanation of As-Prepared CNTs/Fe₃O₄/PANI. In view of the unique and facile synthesis process, the reaction mechanism of CNTs/Fe₃O₄/PANI nanocomposites is schematically illustrated in Figure 4. It is well known that treating the CNTs with sulfuric acid could create considerable functional groups such as carboxyl or carbonyl on the outside surface of CNTs which became negatively charged. The large number of the carboxylic acid or carbonyl groups on the outside of the CNTs could catch

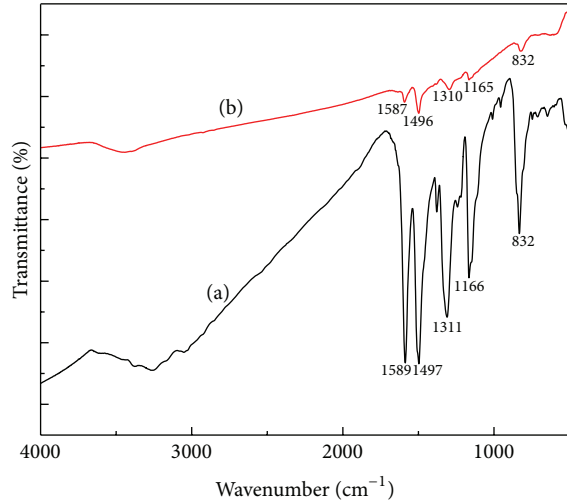
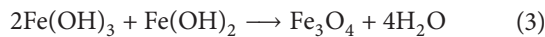
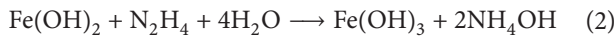
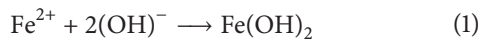


FIGURE 3: FTIR spectra of the (a) PANI and (b) CNTs/Fe₃O₄/PANI.

and strongly bond with Fe²⁺ ions in NH₂Fe(SO₄)₂ · 6H₂O solution through electrostatic attraction. Then, after the addition of N₂H₄ solution, many primary Fe²⁺ ions further interacted with N₂H₄ into Fe³⁺ ions under alkaline condition (PH = 11–13). In the following long-drawn reflux process, large quantities of obtained Fe³⁺ ions and incipient unreacted Fe²⁺ ions went through intensive coprecipitation reaction. Finally the Fe₃O₄ magnetic nanoparticles formed and adhered on the surface of CNTs driven by the minimization of surface energy. The OH⁻ ions play an important role in the formation of Fe₃O₄ magnetic nanoparticles. When NH₃ · H₂O creating OH⁻ ions took place in the aqueous solution, the OH⁻, as an inorganic ligand, would first interact with Fe²⁺ forming Fe(OH)₂. This will promote the further interaction between Fe(OH)₂ and N₂H₄ so that the obtained Fe(OH)₃ could combine with Fe(OH)₂ under the favourable OH⁻ condition. The reaction processes can be briefly described as



Then, the in situ polymerization happened in the quick process, which seemingly improved the distribution of Fe₃O₄ magnetic nanoparticles, getting the fine CNTs/Fe₃O₄/PANI nanocomposites (Figure 1(a)).

4.5. Microwave Absorption Theory. For the microwave absorption properties, the samples were first disposed by mixing with paraffin wax in an ether solution, followed by evaporating the solvent. The dried samples were collected and compressed into a toroidal shape. The reflection loss (RL) values of CNTs/Fe₃O₄/PANI and CNTs/Fe₃O₄ composites were calculated using the relative complex permittivity and permeability at a given frequency and thickness layer

according to the transmit line theory, which is summarized as the following equations [25]:

$$Z_{\text{in}} = \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left[-j \frac{2\pi}{c} \sqrt{\mu_r \epsilon_r} f d \right], \quad (4)$$

$$\text{RL (dB)} = -20 \log_{10} \left| \frac{Z_{\text{in}} - 1}{Z_{\text{in}} + 1} \right|, \quad (5)$$

where ϵ_r and μ_r are the relative complex permittivity and permeability of the composite medium, respectively, f is the frequency of the microwave in free space, c is the velocity of light, d is the absorber thickness, and Z_{in} is the input impedance of the absorber.

4.6. The Comparison of Microwave Absorption Properties between CNTs/Fe₃O₄/PANI and CNTs/Fe₃O₄. Figure 5 shows the reflection loss characteristics curve of the CNTs/Fe₃O₄/PANI and CNTs/Fe₃O₄ samples under the same paraffin wax weight ratio (20 wt%) condition, which was calculated by the previous equations (4) and (5). In this calculation, d was considered to be 2 mm. The samples prepared with PANI exhibit the largest reflection loss of -48 dB at 12.9 GHz, which is in an interval width of 5.2 GHz with losses all below -20 dB. The samples prepared without PANI have a maximum reflection loss of -44 dB at 13.1 GHz with about 5.5 GHz bandwidth below -20 dB. In the comparison of CNTs/Fe₃O₄/PANI with CNTs/Fe₃O₄, the CNTs/Fe₃O₄/PANI samples with PANI have the largest reflection loss of -48 dB, which is higher than that of samples without PANI, while the frequency region (below -20 dB) between the two is approximated. These results indicate that the samples prepared with PANI have relative better absorption properties than those of samples without PANI. Normally, microwave absorption depends on the impedance match conditions of the interfaces between the fillers and air [26]. Thus, the existence of PANI coatings with a different electrical conductivity and dielectric properties could change the impedance between CNTs and air, leading to CNTs/Fe₃O₄ microwave absorption performance superior to CNTs/Fe₃O₄/PANI composites. However, the results display the deviation which can be attributed to the dispersion of the CNTs/Fe₃O₄/PANI samples, which is better than that of the samples prepared without PANI. It is further proved that the dispersion of the CNTs/Fe₃O₄ nanocomposites is poor, and the orientation of the deposition of Fe₃O₄ nanoparticles on the surfaces of CNTs is difficult to control. Also, it is worth noting that the reflection loss results of both are much more effective than those of many naked CNT composites in previous reports [27, 28].

4.7. The Effects of Several Factors on Wave Absorption Properties of CNTs/Fe₃O₄/PANI. Figure 6(a) presents the experimental results of the reflection loss versus frequency for CNTs/Fe₃O₄/PANI wax composites with the increase of paraffin wax weight ratio from 10 to 25 wt% in the range of 2–18 GHz. The CNTs/Fe₃O₄/PANI composites with a paraffin wax weight ratio of 10 wt% show weak wave-absorbing ability,

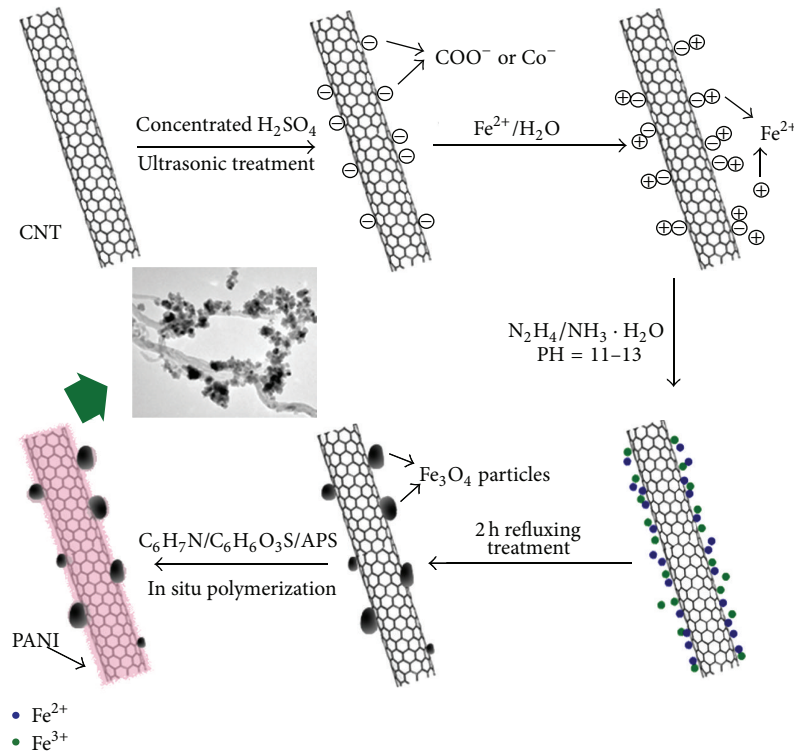


FIGURE 4: The possible formation process of the CNTs/Fe₃O₄/PANI nanocomposites.

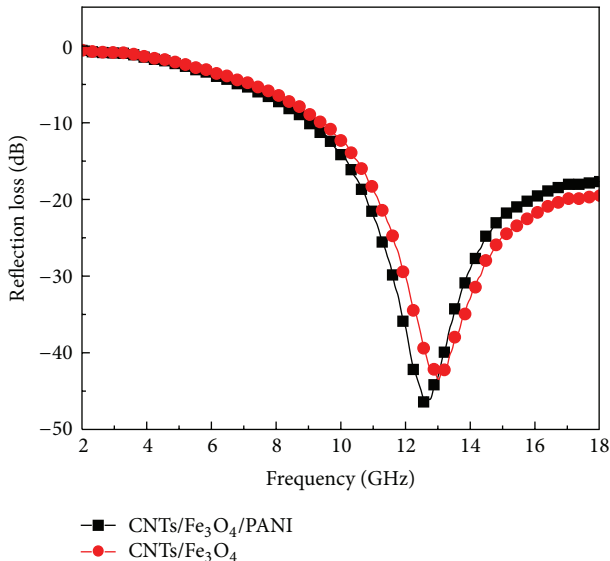


FIGURE 5: Microwave reflection loss curves of CNTs/Fe₃O₄/PANI wax composites and CNTs/Fe₃O₄ wax composites.

and the absorption peak increases to 33 dB at paraffin wax weight ratio of 15 wt% and then reaches the maximum value of 48 dB at the paraffin wax weight ratio of 20 wt%. But with further increasing paraffin wax weight ratio, the wave-absorbing ability decreases. Also, it is clearly seen that the peak position moves to lower frequencies with increasing

paraffin wax weight. In addition, we also explored the influence of absorber thickness on wave-absorbing performance by calculation. The absorber thickness (d) was considered to be 1, 1.5, 2, 2.5, 3, 3.5, and 4 mm, respectively. The evaluated reflection loss of CNTs/Fe₃O₄/PANI composites coupled with different thicknesses is shown in Figure 6(b). It is obviously exhibited that the reflection loss peaks shift from higher to lower frequency as the thickness increases, associated with quarter-wavelength attenuation [29]. Furthermore, the CNTs/Fe₃O₄/PANI composites demonstrate excellent microwave absorption performance with wide absorption bandwidth in an effective range of thickness. In the investigated region, the maximum reflection loss peak achieves up to 48 dB (2 mm in thickness). These results above are of importance since the absorption peak frequency ranges of the CNTs/Fe₃O₄/PANI composites can be tuned easily by changing the wax weight ratio and absorber thickness, and thus a broadband and effective absorption design could be achieved using CNTs/Fe₃O₄/PANI composite nanomaterials.

5. Conclusions

In summary, we have developed a simple method to synthesize the CNTs/Fe₃O₄/PANI composite nanostructures. The Fe₃O₄ magnetic nanoparticles have the diameters of 3–5 nm. The Fe₃O₄ nanoparticles were strongly attached to the surface of CNTs by the action of PANI. The CNTs/Fe₃O₄/PANI composites reveal super microwave absorption properties and possess a maximum reflection loss of -48 dB at 12.9 GHz,

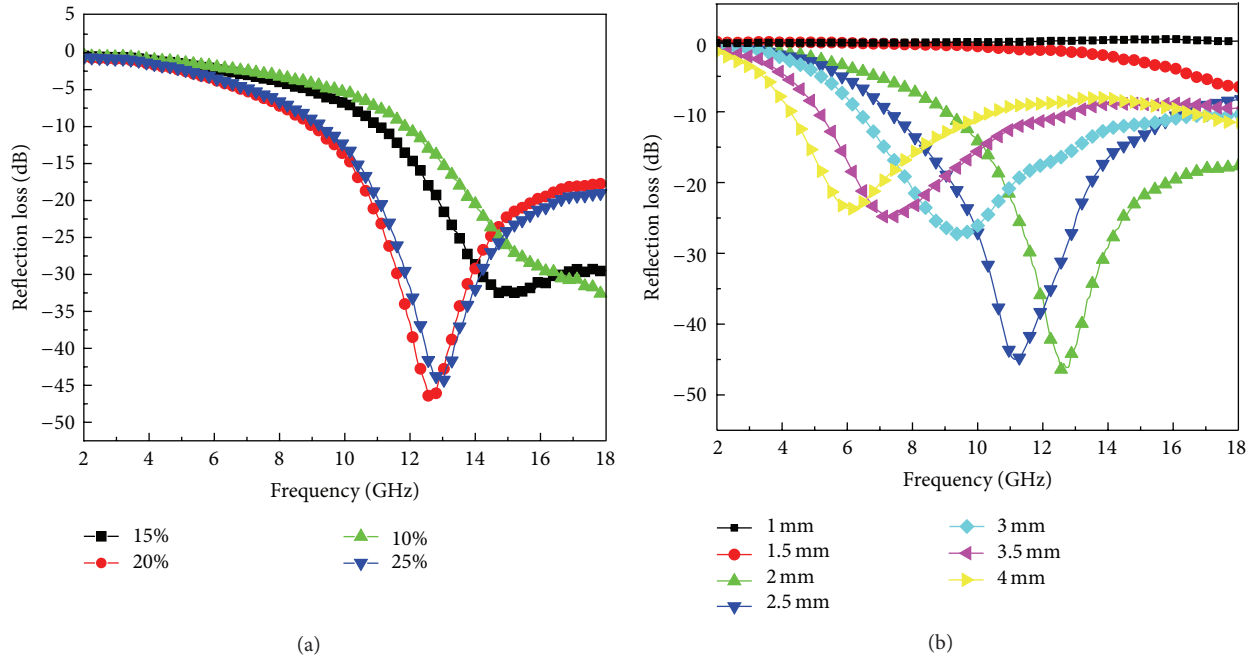


FIGURE 6: Microwave reflection loss curves of CNTs/Fe₃O₄/PANI wax composites with different paraffin wax weight ratio (a) and different thickness (b).

and the bandwidth below -20 dB is more than 5 GHz, which is much higher than many CNT-based samples. In addition, the absorption peak frequency ranges of the CNTs/Fe₃O₄/PANI composites can be tuned by changing the wax weight ratio and its thickness. All the results indicate that the CNTs/Fe₃O₄/PANI composite nanomaterials may be attractive candidate materials for microwave absorption applications.

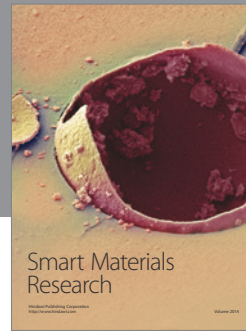
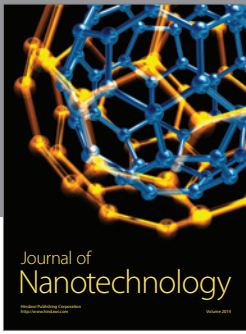
Acknowledgment

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