

Preparation of nanocarbon and metal oxide based composite material for X-band electromagnetic absorption

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ABSTRACT

Electromagnetic waves are believed to be highly effectively absorbed by composite materials based on nanocarbon and magnetic oxides. In this study, NiFeMgO_x multi-metal oxide nanoparticles were prepared by homogeneous precipitation process and then, CNT fibers were chemically vapor deposited onto NiFeMgO_x to obtain the CNT/NiFeMgO_x material. SEM, XRD, and BET techniques were used to characterize the composition of composite materials, and electromagnetic properties such as conductivity, coercivity, and saturation magnetic moment were also investigated. The findings of the study indicated that CNT fibers grew effectively on the surface of NiFeMgO_x nanoparticles. Multi-metallic oxide composed of NiO, FeO and MgO covered with highly graphitized CNTs fibers. The resulting composite material exhibited a concentrated pore distribution ranging from 10 to 50 nanometers and a specific surface area of 90 m²/g. The micro-structured material includes hollow carbon nanotube (CNT) fibers measuring approximately 50 nm in diameter and 2 nm to 4 nm in wall thickness. The CNTs/NiFeMgO_x material showed low coercivity (H_c = 1500 Oe) and high magnetic saturation (M_s = 11.5 emu/g), which makes it a great candidate for the fabrication of materials that absorb electromagnetic radiation. On the basis of CNT/NiFeMgO_x composites, magnetic powder, carbon black, and adhesives, electromagnetic wave-absorbing materials are produced. The results suggest that an X-band material with a thickness of 1.5 mm and an adhesive content of 20% may successfully reduce electromagnetic wave reflection by up to to be -35 dB. The CNTs/NiFeMgO_x composite material has a lot of opportunities to develop to a novel electromagnetic wave adsorbent material system with numerous outstanding characteristics.¹⁻²¹

Key words: CNT/NiFeMgO_x, composite material, electromagnetic absorption

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History

- Received: 19-9-2023
- Accepted: 29-12-2023
- Published Online: 31-3-2024

DOI :

<https://doi.org/10.32508/stdjet.v6iS13.1203>



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INTRODUCTION

Electromagnetic-absorbing materials (EMA) are widely used in instrumentation, technical inspection, communication, and medical equipment¹. In the field of defense, EMA is one of the most effective means to prevent radar detection of flying devices. They make it easier for aircraft and missiles to move through the gaps of enemy air defenses and anti-missile lines and protect fixed targets on the ground and at sea from radar detection by the enemy². Therefore, the research and fabrication of electromagnetic absorbing materials have attracted great research attention.

In 1991, carbon nanotubes (CNTs) were initially found by Iijima. Since the discovery of CNTs, substantial progress has been made in the development of applications for coatings that absorb electromagnetic radiation. The coatings have good ability to absorb electromagnetic waves, which can be applied as camouflage materials in the fields of security, medical, electronics and military defense³.

As we know, electromagnetic waves consist of two components: an electric field and a magnetic field. Materials can absorb electromagnetic waves through different mechanisms depending on their properties. Typically, the magnetic component of an electromagnetic wave interacts with a magnetic material, whereas the electric field component interacts with dielectric materials. CNTs absorb electromagnetic waves mainly based on the dielectric loss mechanism. Because of the lack of magnetic attenuation ability and advantageous dispersion, it is extremely difficult to produce great absorption efficiency for single-component CNTs absorbent. CNTs were combined with other loss materials to improve permeability and interface contact in order to achieve a greater absorption effect. Actually, CNTs-based metal composites are mainly concerned with developing novel nanomaterials by fabricating magnetic nanoparticles on CNTs to satisfy impedance matching while developing special microstructures, whereas multi-component alloys are frequently attached to

Cite this article : Ha B Q, Nguyen T M, Nguyen C V, Trinh H D, Nguyen H T, Ngo H V, Le H T, Le T M. Preparation of nanocarbon and metal oxide based composite material for X-band electromagnetic absorption. *Sci. Tech. Dev. J. – Engineering and Technology* 2024; 6(S13):46-52.

the surface of CNTs. The transition metal oxide like Fe_3O_4 ⁴, Co_3O_4 ⁵, CoFe_2O_4 ⁶, NiFe_2O_4 ⁷, are capable of absorbing electromagnetic waves on the magnetic loss mechanism, and when combined with CNTs, it is possible to obtain electromagnetic wave-absorbing materials according to both above-mentioned mechanisms. SWCNTs and CoFe_2O_4 nanocrystal were combined by Li et al., and the resulting composite material had an RL value of -30.7 dB at 12.9 GHz⁸. Cao and coworkers effectively created the $\text{Fe}_3\text{O}_4/\text{MWCNTs}$ nanocomposite, which exhibits exceptional microwave absorption capability and wide absorption bandwidth^{9,10}. Reduced $\text{GO}/\text{MWCNTs}/\text{ZnFe}_2\text{O}_4$ was easily fabricated by Shu et al. using a 3D conductive network, and it exhibits a minimum RL value of -22.2 dB at the Ku band with a thickness of just 1.0 mm¹¹. Using a simple one-step process, Shu et al. developed a 3D net-like $\text{MWCNTs}/\text{ZnFe}_2\text{O}_4$ hybrid with a mass of conductive networks¹².

Here in, we described a simple one-step CVD method for creating high-performance EMAs made of hybrid composites of CNTs and NiFeMgO_x nanoparticles. The results showed that the $\text{MWCNTs}/\text{NiFeMgO}_x$ hybrid composites were much better at absorbing electromagnetic waves (EMWs), with less reflection loss and a wider absorption bandwidth. Additionally, it explained the potential mechanism of EMW absorption as well as the synergistic effect and interfacial interactions between magnetic NiFeMgO_x microspheres and carbon nanotubes.

MATERIALS AND METHODS

Chemicals and Materials

$\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Mg}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$, glycine were supplied by Fisher Chemical Co. Methane were commercially available from Vietnam Pure Gases JSC.

Experimental methods

Preparation of $\text{CNT}/\text{NiFeMgO}_x$ composites

A mixture of 1 mol $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, and 1 mol $\text{Ni}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, 1 mol $\text{Mg}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$ and 20 g of glycine were dissolved in 20 mL of water. The solution was heated above 50°C and magnetically stirred for 30 minutes. Place the above mixture in an inert gas furnace and heat it to 500 °C at a rate of 5 °C per minute. Continue maintaining this temperature for 60 minutes to obtain NiFeMgO_x oxide.

$\text{CNT}/\text{NiFeMgO}_x$ composite was fabricated by pyrolysis of methane gas on NiFeMgO_x . Place the crucible containing 1g of NiFeMgO_x inside the CVD furnace.

N_2 was blown into the furnace for 30 minutes at a rate of 100 mL/min. Heat the mixture at a rate of 5°C per minute to reach 700°C gradually. Switch the gas from N_2 to methane gas and maintain the methane gas rate of 100 mL/min for 10 minutes. The results obtained were $\text{CNT}/\text{NiFeMgO}_x$ products with a CNT yield of 3g CNT/1g NiFeMgO_x .

Characterization of $\text{CNT}/\text{NiFeMgO}_x$

Using X-ray Bruker D8-Advance diffractometer with a $\text{Cu K}\alpha$ anode ($\lambda = 0.1542$ nm) to characterize the crystal structure of materials with operating condition of 40 kV and 30 mA and the angle range 2θ from 10° to 70° with a step size of 0.05° per step. The specific surface area and porosity distribution were investigated by N_2 isotherm adsorption-desorption method on the Tri Start 3000 at 77 K and degassing temperature of 200 C for 5 h.

Field emission scanning electron microscopy (FE-SEM; HITACHI S-4800) at an accelerating voltage of 15 kV and High-resolution transmission electron microscopy (HRTEM; JEM 2100) at an acceleration voltage of 200 kV were used to characterized the structural properties of materials.

Electromagnetic Properties Measurement

The electromagnetic wave absorbent material is made on the basis of $\text{CNT}/\text{NiFeMgO}_x$ composites, with the ratio of components as follows: 100 parts by weight of chlorinated polyvinyl chloride resin and $\text{CNT}/\text{NiFeMgO}_x$, and 50 parts of cyclohexanol solvent. The above ingredients are ground by a planetary ball mill for 1 hour and then evenly coated on a 200 mm by 200 mm aluminum sheet. The coating thickness is 150 nm. The electromagnetic properties were measured at a frequency of 8-12 GHz on a keysight RF analyzer.

RESULTS AND DISCUSSION

Figure 1 exhibit the N_2 gas adsorption-desorption isotherm and pore distribution of $\text{CNT}/\text{NiFeMgO}_x$ composite. The N_2 gas adsorption-desorption curve can be classified into type II model which is characteristic for macro-porous materials. The N_2 adsorption volume at the low P/P° area show that a significant overlapping of monolayer adsorption and the beginning of multilayer adsorption. The increase of adsorption volume at the $P/P^\circ=1$ due to macro-porous pore and multilayer N_2 diffusion and adsorption behavior. The analysis of the specific surface area of $\text{CNT}/\text{NiFeMgO}_x$ showed quite high value (90 m^2/g). The pore distribution diagram demonstrates that the

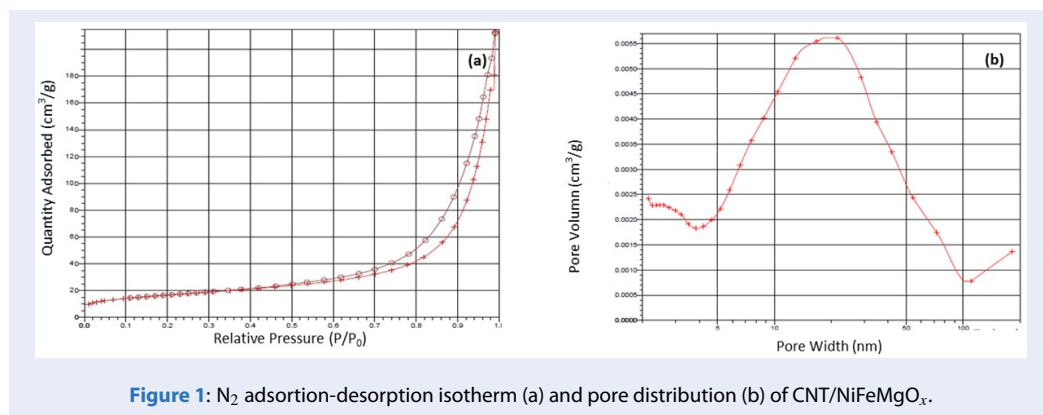


Figure 1: N₂ adsorption-desorption isotherm (a) and pore distribution (b) of CNT/NiFeMgO_x.

material has minimal pores of over 100 nanometers in size. The porosity of these samples primarily originates from openings ranging from 10 to 50 nm.

Illustrated in Figure 2 are the SEM and TEM images of CNT/NiFeMgO_x. The SEM image shows that the CNTs were successfully fabricated on NiFeMgO_x nano particles. The average diameter of CNTs was about 50 nm. It can be clearly observed that the NiFeMgO_x nanoparticles are well-distributed on the surface of CNTs with some degree of the aggregation. The majority of NiFeMgO_x nanoparticles were approximately 30 nm in diameter. The semitransparent of the CNTs implies that the CNT's wall consist of several layers with the thickness of 2-4 nm.

The results of X-ray diffraction (XRD) were employed to analyze the properties of the phase composition of CNT/NiFeMgO_x. The XRD patterns of NiFeMgO_x nano particles and CNT/NiFeMgO_x are shown in Figure 3a and Figure 3b, respectively. XRD pattern of NiFeMgO_x shows that the crystallite peaks appearing at $2\theta = 30.1^\circ, 35.0^\circ, 37.2^\circ, 43.2^\circ, 53.5^\circ, 56.9^\circ, 62.6^\circ, 75.2^\circ,$ and 78.7° are characteristic of FeO, MgO and NiO as well as the formation of rock salt NiFeMgO_x solid solution¹³.

The diffraction peaks mentioned above can also be seen on the CNT/NiFeMgO_x XRD pattern, but their intensity has been noticeably reduced. The diffraction peak at position $2\theta = 26^\circ$ is typical for the graphitic carbon of CNTs. XRD result demonstrates that the produced CNTs growth on NiFeMgO_x exhibits high graphitization degree^{14,15}.

The magnetization curve (Figure 4) displays the relation of the mass magnetization M with the magnetic field H_c of CNT/NiFeMgO_x composite. The curve of is similar to others composites reported in the literature^{16,17}. The curve is typical for soft magnetic materials with a large value of saturation magnetization ($M_s=11.5$ emu/g) and a low coercivity ($H_c=1500$ Oe).

According to the magnetic property measurements, CNT/NiFeMgO_x is suitable for the production of electromagnetic absorption materials¹⁰.

The electromagnetic wave absorption characteristic of the material is shown by the attenuation of wave reflection intensity at frequencies from 8 to 12 GHz. It is clear that the samples attained reflection intensity has been attenuated between 10 and 11 GHz. Samples with 15%, 20%, and 25% CPVC compared to CNT/NiFeMgO_x demonstrated the best attenuation of 32 dB, 35 dB, and 28 dB. With a film-forming ratio of 20%, the material can absorb up to 99.9% of electromagnetic waves with wide absorption bandwidth. The optimal amount of the film-forming agent allows consistent film formation without having a significant impact on the electromagnetic wave absorption properties of CNT/ NiFeMgO_x

Compared to previous reports on CNT based material for electromagnetic wave absorption application, this result was quite favorable (Table 1). Using the CVD technique, CNT is directly deposited onto a metal oxide substrate to fabricate the material. As a result, the material possesses an interlaced, multi-layer porosity structure with uniformly sized CNT fibers. Due to the combination of the magnetic properties of metal oxide and the electrical conductivity of CNT, CNT/NiFeMgO_x showed good electromagnetic wave absorption efficiency^{3,18}.

CONCLUSIONS

CNT/NiFeMgO_x composite was fabricated by pyrolysis of petroleum gas process at high temperature and neutral atmosphere. The average diameter of CNTs was about 50 nm. The NiFeMgO_x nanoparticles with approximately 30 nm in diameter are well-distributed on the surface of CNTs with some degree of the aggregation. The obtained composite exhibit soft magnetic materials properties with a large

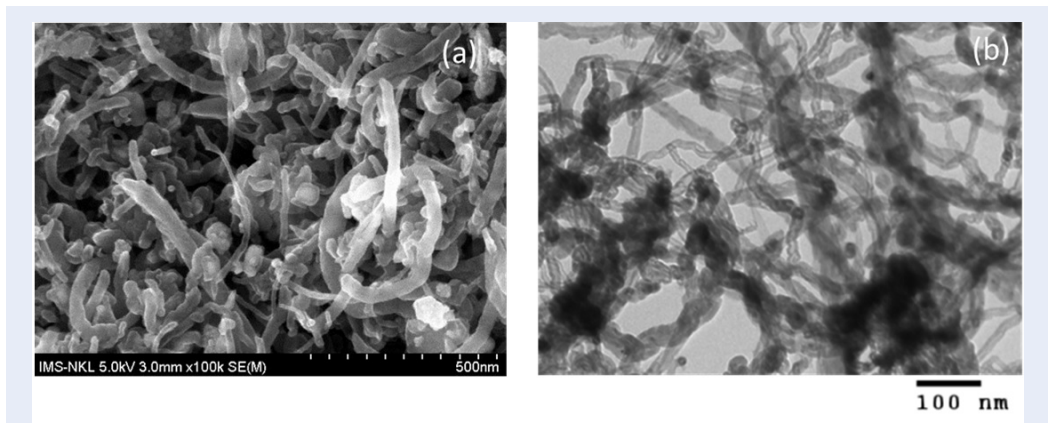


Figure 2: SEM image (a) and TEM image (b) of CNT/NiFeMgO_x.

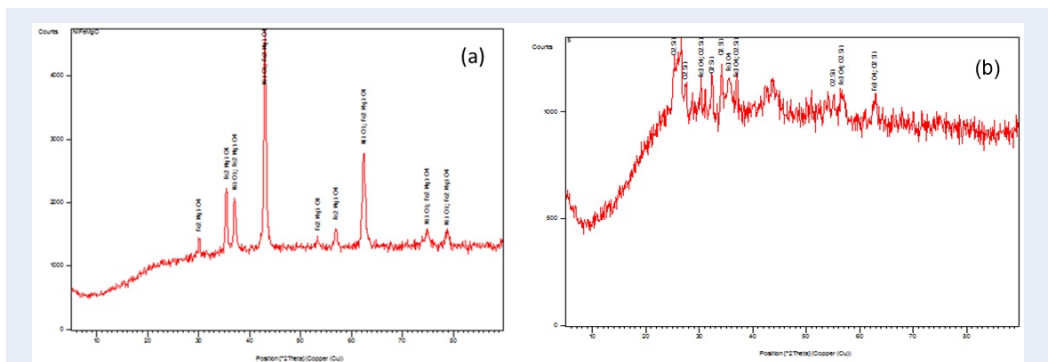


Figure 3: XRD pattern of NiFeMgO_x (a) and CNT/NiFeMgO_x (b)

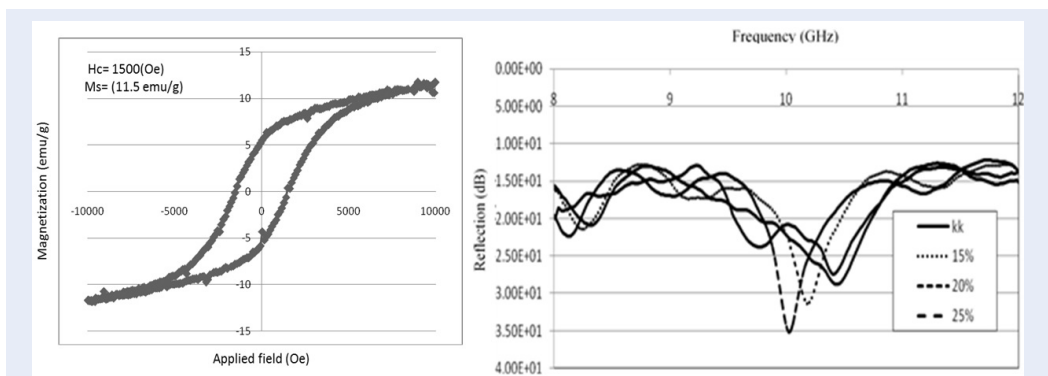


Figure 4: Magnetization curve at 298 K and Electromagnetic reflection of CNT/NiFeMgO_x

Table 1: Comparisons of electromagnetic wave absorption effects of CNT based composites

Materials	Electro-magnetic wave absorption	References
10 μm CNTs/epoxy composite	18 dB (4-12 GHz)	14
4 mm of CNT/SiCf composites	62.5 dB (2-18 GHz)	15
3.5 mm CNTs paraffin composite	-22 dB (0-18 GHz)	16
2.0 mm Fe ₃ O ₄ @Ti ₃ C ₂ Tx/CNTs composite	40.1 dB (2-18 GHz)	17
2.0 mm CNT/NiFeMgOx composite	35 dB (8-12 GHz)	This work

value of saturation magnetization ($M_s=11.5$ emu/g). The CNT/NiFeMgOx exhibited high of microwave absorption performance with RL of - 35 dB.

ACKNOWLEDGMENT

The research funding from Science and Technology Development Program through the Institute of Chemistry and Materials was gratefully acknowledged.

DECLARATION OF COMPETING INTEREST

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

B.Q.Ha: Material preparation, data interpretation, analysis and writing-original draft preparation; H.T.Nguyen, H.V.Ngo, H.D.Trinh: Data analyzing, writing-reviewing and editing; C.V.Nguyen, H.T. Le: Discussion and data curation; T. M. Nguyen; T.M. Le: Review and editing, supervision.

All authors have read and approved the final manuscript.

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Vật liệu composit hấp thụ sóng điện từ băng X trên cơ sở nano cacbon và oxit kim loại

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TÓM TẮT

Vật liệu composit trên cơ sở nano cacbon và oxit kim loại từ có khả năng hấp phụ tốt sóng điện từ. Trong nghiên cứu này, hạt oxit đa kim loại nano oxit NiFeMgO_x được chế tạo bằng phương pháp đồng kết tủa và sợi CNT được phủ lên bề mặt các hạt bằng phương pháp lắng đọng hơi hóa học, thu được vật composit CNTs/NiFeMgO_x. Sử dụng các phương pháp hiện đại như SEM, XRD, BET để khảo sát thành phần cấu trúc và đặc tính bề mặt của vật liệu, ngoài ra đặc tính từ cũng được nghiên cứu làm rõ. Kết quả nghiên cứu cho thấy sợi CNTs đã mọc thành công trên bề mặt hạt nano NiFeMgO_x. Oxit đa kim loại dạng tổ hợp giữa NiO, FeO và MgO được bao phủ bởi các sợi CNTs có độ graphit hóa cao. Vật liệu composit thu được có diện tích bề mặt riêng là 90 m²/g, phân bố lỗ xốp tập trung trong khoảng 10 nm- 50 nm. Vật liệu có vi cấu trúc là các sợi CNTs rỗng có đường kính khoảng 50 nm, độ dày thành ống khoảng 2 nm- 4 nm. Vật liệu CNTs/NiFeMgO_x có đặc tính từ mềm với từ độ bão hòa cao đạt Ms=11.5 emu/g và lực kháng từ thấp Hc=1500 Oe, rất phù hợp cho chế tạo vật liệu hấp thụ sóng điện từ. Vật liệu hấp thụ sóng điện từ được chế tạo với thành phần chính là composit CNT/NiFeMgO_x, than đen, chất kết dính. Kết quả cho thấy, khi sử dụng 20% hàm lượng chất kết dính, vật liệu vật liệu dày 1,5 mm cho độ suy giảm phản xạ sóng điện từ tới 35 dB trong dải băng tần X. Kết quả nghiên cứu cho thấy vật liệu composit CNTs/NiFeMgO_x có nhiều triển vọng để phát triển thành hệ vật liệu hấp thụ sóng điện từ mới với nhiều đặc tính vượt trội.

Từ khóa: CNT/NiFeMgO_x, vật liệu composit, hấp thụ sóng điện từ

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Lịch sử

- Ngày nhận: 19-9-2023
- Ngày chấp nhận: 29-12-2023
- Ngày đăng: 31-3-2024

DOI : <https://doi.org/10.32508/stdjet.v6iS13.1203>



Bản quyền

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Trích dẫn bài báo này: Bảng H Q, Tường N M, Cành N V, Hoàn T D, Hòa N T, Hoàn N V, Hiếu L T, Trí L M. **Vật liệu composit hấp thụ sóng điện từ băng X trên cơ sở nano cacbon và oxit kim loại.** *Sci. Tech. Dev. J. - Eng. Tech.* 2024, 6(S13):46-52.