Fabrication of cobalt filled multi-walled carbon nanotubes/polyurethane composite for microwave absorption

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Received: 14 February 2020 / Accepted: 23 October 2020 / Published online: 20 November 2020
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Abstract

This research work describes the design and method of development of microwave absorber. This study was conducted for analysis of reflection loss performance with the magnetic modifications of Multi-Walled Carbon Nanotubes (MWCNTs). Cobalt filled Multi-Walled Carbon Nanotubes composites were prepared by three step method. Composites were developed with varying weight percentage of Cobalt Sulphate and Multi-Walled Carbon Nanotubes. The morphology, elementary analysis and absorbing properties of Cobalt filled Multi-Walled Carbon Nanotubes composites were studied by FESEM, EDX and Vector Network Analyzer. The maximum reflection loss is observed for (50% MWCNT and 50% Cobalt Sulphate) is -30.22 dB at 11.8 GHz and the maximum bandwidth window is available for (40% MWCNT and 60% Cobalt Sulphate) is 3.9 GHz in the frequency range of 8–13 GHz with 3 mm thickness, which can be credited to synergistic effect of improved matched impedance and greater microwave attenuation properties of the absorber. The combined usage of dielectric loss and magnetic loss absorber design shows great diversity and can be a promising candidate for designing high performance microwave absorbing materials.

Keywords Multi-walled carbon nanotubes (MWCNTs) · Wet chemical method · Microwave absorption · Reflection loss

1 Introduction

Radar detection reduction technique protects an airplane against radar cross section (RCS) detection which becomes vital in modern high-performance combat technology equipments. RCS can be reduced by altering the shapes of airplanes, radar absorbing materials (RAM). The aim of shaping is to alter the system designs to reduce RCS but shaping has some limitation from the view of manufacturing industry because they cannot compromise with system designs just for the sake of RCS reduction. Despite of this, the developers may coat delicate parts of the target with Radar Absorbing Material. The Radar Absorbing Materials are developed to absorb electromagnetic waves so as to reduce the reflected waves and RCS reduction [1]. Magnetic or dielectric particles are essentially used for

the microwave absorption materials. Magnetic or metal particles have high relative gravity and challenging construction which constrained their real-world applications. Therefore, there is a requirement for an effective electromagnetic wave absorbing material which should be cost-effective, flexible, lightweight and efficient in absorption in a wide range of frequencies. Nanoparticles composites have great fascination for fabrication of electromagnetic wave absorbing materials in a wide frequency spectrum due to their various exceptional chemical, physical and electrical characteristics. The unique properties and an excellent structure of carbon nanotubes (CNTs) causes extensive studies for many prospective EMI shielding applications [2–4]. Carbon nanotubes (CNTs) are assembled by carbon atoms with covalent bonding between carbon–carbon atoms, are nano scale structures [5].

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CNTs are discovered by Iijima in 1991. Since its discovery, CNTs have become the main focus of analyzers all over the world because of its incredible physical properties due to strong atomic bonding. Carbon nanotubes are categorized into two categories, Single-Walled Carbon Nanotubes (SWCNTs) and Multi-Walled Carbon Nanotubes (MWCNTs). CNTs also have high electric conductivity which enhances electromagnetic wave absorption [6–10]. Ferromagnetically modified CNTs have gained intensive attention towards the microwave absorbing performance of nanocomposites.

Many researches had endeavored to encapsulate the CNTs with ferromagnetic material. Encapsulations of ferromagnetic materials like Co, Ni, Fe and Zn in the CNTs are greatly fascinating nanostructure materials with magnetic and dielectric properties. There are some analysis reported on MWCNTs with different composite materials which depicts the microwave absorption properties of the composites [11–15]. Jie Sun et al. prepared a copper cobalt nickel ferrite/graphene oxide/polyaniline tri-composite and investigated in the frequency range of 2–18 GHz. When the usage of tri-composite is about 40%, it has a maximum reflection loss of –33 dB at 10.8 GHz with 2 mm thickness [16]. Qilei Wu et al. synthesized the cobalt doped porous carbon composite with a low-dielectric amorphous carbon/Zn shell shows maximum reflection loss of –43.97 dB at 10.86 GHz with absorption bandwidth of 4.1 GHz [17]. Pei-Yan Zhao et al. fabricated Ni/MWCNTs composites with 20 wt% fillers shows reflection loss of –46.85 dB at 6.56 GHz with 3.2 GHz bandwidth and 3.7 mm thickness in frequency range of 2–18 GHz [18]. Wei Chen et al. manufactured the double-layered composites and characterized in X-band. The maximum reflection loss of double-layered composites is –45.7 dB with 4.2 GHz bandwidth and 1.8 mm thickness [19]. Yiru Li et al. fabricated the Ni/MWCNTs hybrid structure showed the minimum RL of Ni/MWCNTs (20 wt%) hybrids was –40.65 dB at 11.2 GHz with 3.6 GHz bandwidth and 1.8 mm thickness in 2–18 GHz frequency range [20]. Zhen-Jie Guan et al. constructed the sandwich-like cobalt/reduced graphene oxide/cobalt composites showed maximum reflection loss was –61 dB at 11.1 GHz with 4 GHz bandwidth and 2 mm thickness in 2–18 GHz frequency range [21]. Mei Wu et al. synthesized the Graphene oxide/CNT/Co_xFe_{3–x}O₄ (x = 0.375) ternary nanocomposites has reflection loss value of –71.3 dB with the bandwidth of 2.52 GHz and thickness of 7.92 mm in 2–18 GHz frequency range [22]. Xiaogang Su et al. investigated the CoFe₂O₄/FeCo/Graphite hybrid composites at filling ratio of 50 wt% showed, maximum reflection loss of –54.3 dB at 1.2 mm thickness and 4.08 GHz bandwidth at 1.3 mm thickness in 2–18 GHz frequency range [23]. Zhen Xiang et al. synthesized the γ-Fe₂O₃/MWCNTs hollow nanosphere composites with a filling ratio of 20 wt%

achieved reflection loss of –63.6 dB at 1.4 mm thickness and 5.2 GHz bandwidth in 2–18 GHz frequency range [24]. Li Peibo et al. prepared the Co/C (70%), Ba-hexaferrite (29%), MWCNTs (1%) composites shows maximum reflection loss was –26 dB at 12.4 GHz with 4.2 GHz bandwidth and 2.5 mm thickness in the X band [25].

The main objective of this work was to exhibit an easiest procedure for the fabrication of Cobalt filled MWCNTs/Polyurethane composites for microwave absorption. During filling of Cobalt in MWCNTs, the nitrogen atmosphere was required. Nitrogen gas was fed into flasks through syringe needle which was connected with syringe and then balloons. Main principle of this work was to fill the magnetic nanoparticles into the MWCNTs and to study material characteristics i.e. morphology and elemental analysis of nanocomposites. Then Polyurethane was added to Co/MWCNTs nanocomposites, to made the samples harder and to study electrical characteristics i.e. reflection loss performance in the frequency range of 8–13 GHz.

2 Experiment

2.1 Materials

The MWCNTs with 5–20 nm diameter, 2–10 μm length, less than 2% ash contents, more than 97% purity, high thermal and electric conductivity were purchased from Aritech Chemazone PVT. LTD., Kurukshetra, Haryana. Nitric Acid (68% concentration), Cobalt (II) Sulphate, *N,N*-Dimethylformamide (DMF), Polyurethane (PU) were purchased from A Kay Scientific Store, Jalandhar, Punjab.

2.2 Synthesis of composites

2.2.1 Functionalization of MWCNTs

MWCNTs were heated in nitric acid (68%) using water bath refluxer upto 100 °C for 7 h to remove trace impurities. Refluxed mixture was filtered with distilled water using Suction Pump filtration until pH 7. Further it was then dried at 50 °C in hot air oven for 12 h results in functionalized MWCNTs [26–33]. Functionalized MWCNTs were filled with cobalt by wet-chemical method [34] with two different experimental analysis, in which first one includes the varying composition proportion of functionalized MWCNTs and Cobalt Sulphate while the other one includes the varying weight percentage of Cobalt Sulphate solution to develop Cobalt filled MWCNTs nanoparticles composites (Fig. 1).

Fig. 1 Functionalization of MWCNTs **Table 1** Details of varying weight compositions of MWCNTs and Cobalt Sulphate

Sample code	MWCNT (%)	Cobalt Sulphate CoSO ₄ (%)
A	40	60
B	50	50
C	60	40
D	70	30

2.2.2 Analysis 1

In this analysis, MWCNTs were filled with cobalt after successfully functionalization of MWCNTs. Disperse MWCNTs and Cobalt Sulphate of different weight composition in 1000 ml distilled water using magnetic stirrer for 24 h as shown in Table 1. The mixture was filtered and rinsed with distilled water using Suction Pump filtration and dried at 45 °C in hot air oven for 15 h. Then, dried samples were heated for 1 h under the nitrogen atmosphere, by rising its temperature from room temperature to 100 °C using sand bath on hot plate. Then, samples were calcinated for 6 h under the nitrogen atmosphere from room temperature to 450 °C to reduce the metal oxides by the use of muffle furnace (Fig. 2).

2.2.3 Analysis 2

In this analysis, MWCNTs were filled with varying percentage composition of Cobalt Sulphate solution after successfully functionalization of MWCNTs. The functionalized MWCNTs were stirred with CoSO₄ solution (varying wt%) in 200 ml distilled water using magnetic stirrer for 24 h as shown in Table 2. The mixture was filtered and rinsed with distilled water using Suction Pump filtration and dried at 45 °C in hot air oven for 15 h. Then, dried

samples were heated by rising its temperature from room temperature to 100 °C for 1 h under the nitrogen atmosphere using sand bath on hot plate. Then, samples were calcinated from room temperature to 450 °C for 6 h under the nitrogen atmosphere to reduce the metal oxides by the use of muffle furnace (Fig. 3).

2.2.4 Preparation of cobalt filled MWCNT/PU composites

Cobalt filled MWCNTs composites prepared in analysis 1 and 2 were dispersed in *N,N*-Dimethylformamide (DMF) for 6 h using ultra sonication. Then, Polyurethane (PU) were dissolved in DMF in another beaker using magnetic stirring for 6 h. The dispersed MWCNTs and PU were mixed thoroughly using magnetic stirrer for next 6 h [35–37]. The solutions were dried in vacuum oven at 3 kPa (22.5 mm of Hg) for next 6 h until gel formation and then poured into WR-90 waveguide mould of dimensions 22.86 mm (length) × 10.16 mm (width) × 3 mm (thickness) and dried at 35 °C for 20 h in hot air oven (Fig. 4).

Table 2 Details of varying compositions of MWCNTs and Cobalt Sulphate

Sample code	MWCNT (mg)	Cobalt Sulphate CoSO ₄ (%)
P	600	30
Q	500	40
R	400	50
S	300	60

Fig. 2 Cobalt filled MWCNTs for Analysis 1

Fig. 3 Cobalt filled MWCNTs for Analysis 2 **Fig. 4** Cobalt filled MWCNT/PU composites

3 Results and discussion

3.1 Morphology and elemental analysis of cobalt filled MWCNTs composites (analysis 1)

Field emission scanning electron microscopy (FESEM) was used to understand the morphology of the nano-composites. Raw MWCNTs are tubular in structure with the diameter of 5–20 nm, 2–10 μm length. While Functionalized MWCNTs have suspended bonds which were created after acid treatment of MWCNTs. MWCNTs were functionalized to break Carbon–Carbon bonds of MWCNTs and to form C–OH or C–OOH bonds, which were used for further chemical treatments to bond other elements with MWCNTs.

Sample A has 60% Cobalt Sulphate and 40% MWCNT. Figure 5a shows the morphology of MWCNT filled with Cobalt. In Fig. 5a each nanotube contains denser cobalt nanoparticles due to high percentage contents of cobalt as compared to MWCNTs. Figure 6a shows the EDX spectrum and Fig. 6b shows the element analysis, element percentage and atomic percentage of elements present

in sample A, which contains Carbon and Cobalt nanoparticles but there are some other impurities also available i.e. Oxygen, Sodium, Silicon, and Chlorine. Sample A has impurities of Oxygen which arise due to the oxides impurities entrapped in nanoparticles even after the flow of nitrogen gas through them. Sample A has impurities of Silicon due to silicon crucible used for calcination of samples. Sample A has impurities of Sodium and Chlorine due to unwanted impurities present in laboratory, but due to their low weight percentage it doesn't much affect the microwave absorption performance. Sample B has 50% Cobalt Sulphate and 50% MWCNT. Figure 5b shows the morphology of MWCNT filled with Cobalt. In Fig. 5b there is a homogeneous matrix of MWCNTs and Cobalt nanoparticles due to same percentage composition of Cobalt and MWCNTs nanoparticles. Figure 7a shows the EDX spectrum and Fig. 7b shows the element analysis, element percentage and atomic percentage of elements present in sample B which contains Carbon and Cobalt nanoparticles but there are some impurities of Oxygen, Silicon, Chlorine and Iron which arise during the chemical reactions processes and testing of material in laboratory.

Fig. 5 FESEM of Analysis 1: (a) Sample A (b) Sample B (c) Sample C (d) Sample D

Fig. 6 (a) EDX Spectrum of Sample A (b) EDX composition of Sample A

Fig. 7 (a) EDX Spectrum of Sample B (b) EDX composition of Sample B

Fig. 8 (a) EDX Spectrum of Sample C (b) EDX composition of Sample C

Sample C has 40% Cobalt Sulphate and 60% MWCNT. Figure 5c shows the morphology of MWCNT filled with Cobalt. In Fig. 5c, there is lesser number of cobalt nanoparticles present in each carbon nanotube as compared to the morphology of Fig. 5a, b. Figure 8a shows the EDX spectrum and Fig. 8b shows the element analysis, element percentage and atomic percentage of elements present in sample C which contains Carbon and Cobalt nanoparticles with Silicon impurity. Sample D has 30% Cobalt Sulphate and 70% MWCNT. Figure 5d shows the morphology of MWCNT filled with Cobalt. In Fig. 5d, there is least number of cobalt nanoparticles present in each carbon nanotube as compared to the morphology

of Fig. 5a–c. Figure 9a shows the EDX spectrum and Fig. 9b shows the element analysis, element percentage and atomic percentage of elements present in sample B which contains Carbon and Cobalt nanoparticles with Aluminium impurity which arise during the during chemical reactions processes.

3.2 Morphology and elemental analysis of cobalt filled MWCNTs composites (analysis 2)

Sample P has 30% Cobalt Sulphate and 600 mg MWCNT. Figure 10a shows the morphology of MWCNT filled with Cobalt. In Fig. 10a each nanotube contains cobalt

Fig. 9 (a) EDX Spectrum of Sample D (b) EDX composition of Sample D

Fig. 10 FESEM of Analysis 2: (a) Sample P (b) Sample Q (c) Sample R (d) Sample S

Fig. 11 (a) EDX Spectrum of Sample P (b) EDX composition of Sample P

nanoparticles. Figure 11a shows the EDX spectrum and Fig. 11b shows the element analysis, element percentage and atomic percentage of elements present in sample P, which contains Carbon and Cobalt nanoparticles but there are some other impurities are also available i.e. Oxygen, Aluminium, Silicon, Sulphur and Iron. Sample P have impurities of Sulphur due to dispersion of Cobalt Sulphate solution into MWCNTs and on filtration some contents left behind in the Cobalt/MWCNTs nanocomposites, but due to their low weight percentage it doesn't much affects the microwave absorption performance. Sample Q has 40% Cobalt Sulphate and 500 mg MWCNT. Figure 10b shows the morphology of MWCNT filled with Cobalt. Figure 12a

shows the EDX spectrum and Fig. 12b shows the element analysis, element percentage and atomic percentage of elements present in sample Q, which contains Carbon and Cobalt nanoparticles and Oxygen due to oxides atom entrapped in MWCNTs. Sample R has 50% Cobalt Sulphate and 400 mg MWCNT. Figure 10c shows the morphology of MWCNT filled with Cobalt. Figure 13a shows the EDX spectrum and Fig. 13b shows the element analysis, element percentage and atomic percentage of elements present in sample R, which contains Carbon and Cobalt nanoparticles but there are some other impurities are also available i.e. Oxygen, Aluminium, Silicon, and Iron. Impurities like Aluminium, Iron arise during the during chemical

Fig. 12 (a) EDX Spectrum of Sample Q (b) EDX composition of Sample Q

Fig. 13 (a) EDX Spectrum of Sample R (b) EDX composition of Sample R

Fig. 14 (a) EDX Spectrum of Sample S (b) EDX composition of Sample S

reactions processes. Sample S has 60% Cobalt Sulphate and 300 mg MWCNT. Figure 10d shows the morphology of MWCNT filled with Cobalt. Figure 14a shows the EDX spectrum and Fig. 14b shows the element analysis, element percentage and atomic percentage of elements present in sample S, which contains Carbon and Cobalt nanoparticles with Oxygen and Silicon impurities.

3.3 Microwave reflection properties of analysis 1

The reflection loss spectra in the range of 8–13 GHz of composites of analysis 1 are summarized in Fig. 15. From

the curves, it is observed that the maximum reflection loss is observed for sample B is -30.33 dB at 11.8 GHz and minimum reflection loss is observed for sample D is -7.38 dB at 11.8 GHz. Figure 15 shows that Sample A has 3.9 GHz bandwidth from 8.63 to 12.53 GHz frequency range with reflection loss less than -10 dB. Sample B has 3 GHz bandwidth from 9 to 12 GHz frequency range. Sample C has bandwidth 2.45 GHz from 8.75 to 11.2 GHz frequency range. Sample D has bandwidth 1.22 GHz from 9.48 to 10.7 GHz frequency range. The maximum bandwidth window is available for Sample A.

Fig. 15 Reflection Loss performance of composite with wt% of MWCNTs and Cobalt compositions of samples (A, B, C, and D)

Fig. 16 Reflection Loss performance of composite with varying MWCNTs and Cobalt compositions of samples (P, Q, R, and S)

3.4 Microwave reflection properties of analysis 2

The reflection loss spectra in the range of 8–13 GHz of composites of analysis 2 are summarized in Fig. 16. From the curves, it is observed that the maximum reflection loss is observed for sample S is -16.15 dB at 8.03 GHz and minimum reflection loss is observed for sample R is -4.30 dB at 8.85 GHz. Figure 16 shows that Sample Q has 1.87 GHz bandwidth from 9.93 to 11.8 GHz frequency range. Sample S has bandwidth 2.15 GHz from 9.63 to 11.78 GHz frequency range. The maximum bandwidth window is available for Sample S.

3.5 Comparison of microwave reflection properties of analysis 1 and analysis 2

In this analysis, sample A, B, C and D has varying composition of MWCNTs and Cobalt Sulphate in definite proportion with respect to each other and sample P, Q, R, S has varying proportion of Cobalt Sulphate only which is shown in Fig. 17. It has been observed that Sample B has maximum reflection loss of -30.33 dB at 11.8 GHz frequency

Fig. 17 Reflection Loss performance of composite with varying MWCNTs and Cobalt compositions of samples (A, B, C, D, P, Q, R, and S)

with 0.4 GHz bandwidth window. Sample B has 50% MWCNT (dielectric material) and 50% Cobalt Sulphate (magnetic material) composition. So, the maximum reflection occurs due to equal proportion of dielectric and magnetic properties. It has been observed that the maximum bandwidth window is available for Sample A i.e. 3.9 GHz in the frequency range of 8–13 GHz. Sample A has 40% MWCNT (dielectric material) and 60% Cobalt Sulphate (magnetic material) composition. So, the maximum window occurs due to enhanced magnetic properties over dielectric properties.

During the functionalization and filling of cobalt in MWCNTs, microstructure of MWCNTs changed prominently thus more suspending bands and imperfections were existed in Co filled MWCNTs. These suspending bands and imperfections results to more number of scattering of the electromagnetic waves and interfacial electric polarizations, which provide an important tool of microwave absorption. In Co encapsulated MWCNTs the energy levels are split (not continual), because of quantum confine effect, as stated by the Kubo theory. This effect greatly enhances the magnetic and absorbing characteristics of Co. In microwave energy range, the electrons from lower energy level absorbs the photons and jumps to a higher energy level. The MWCNTs and Polyurethane have many interfaces between their layers and outer surface. Therefore, interfacial multipoles between MWCNTs and Polyurethane contribute to the microwave absorption (Table 3).

4 Conclusion

In this work, Cobalt filled MWCNTs/Polyurethane composites were synthesized. The characterizations of Cobalt filled MWCNTs/Polyurethane composites were investigated by FESEM, EDX and VNA in the frequency range

Table 3 Analysis of composite absorber in term of Reflection Loss, Thickness and Bandwidth in the range of 8–13 GHz

Sample code	Thickness of absorber (mm)	Maximum reflection loss (dB)	Frequency for maximum reflection loss (GHz)	Bandwidth (GHz)
A	3	−18.38	9.45	3.9
B	3	−30.33	11.8	3
C	3	−19.55	9.85	2.45
D	3	−12.96	10.10	1.22
P	3	−9.5	11	No BW
Q	3	−14.7	10.85	1.87
R	3	−8.35	12.95	No BW
S	3	−16.15	8.03	2.15

Table 4 Different microwave absorption characteristics of previously reported and present work

Material (wt%)	Matrix	Thickness of absorber (mm)	Maximum reflection loss (dB)	Bandwidth (GHz)	Frequency for maximum reflection loss (GHz)	Reference
Carbon black (50%)	Polyethylene	2.4	−17.5	0.6	10.1	[38]
MWCNTs (2%)	Epoxy resin	2.24	−25	3	10.9	[39]
Co/CNTs (20%)	Epoxy resin	1.8	−20	2.8	8	[40]
Co-C/MWCNTs (25%)	Paraffin	2.5	−28	3.6	10	[41]
Co/CNT (50%)	Paraffin	1	−21.84	1.6	12.2	[35]
CoFe ₂ O ₄ /FeCo/Graphite (50%)	Epoxy resin	1.7	−37	1.5	11.9	[23]
Co/C (70%), Ba-Hexaferrite (29%), MWCNTs (1%)	Paraffin	2.5	−26	4.2	12.4	[25]
Graphene oxide/CNTs/Co _x Fe _{3-x} O ₄ , x = 0.6 (80.99%)	Epoxy resin	8.50	−18	1	12	[22]
MWCNTs (20%), CoSO ₄ (30%)	Polyurethane	3	−18.38	3.9	9.45	This work
MWCNTs (25%), CoSO ₄ (25%)	Polyurethane	3	−30.33	3	11.8	This work

of 8–13 GHz. The maximum reflection loss achieved was −30.22 dB at 11.8 GHz with 3 mm thickness and 0.4 GHz bandwidth at 50% MWCNT and 50% Cobalt Sulphate composition. The maximum bandwidth was 3.9 GHz with 3 mm thickness at 40% MWCNT and 60% Cobalt Sulphate composition. The reflection loss enhancement of the Co filled MWCNTs/Polyurethane composites owing to the involvement of both Co and MWCNTs, which provides combine effect of dielectric losses, magnetic losses, improved matched impedance and larger microwave attenuation properties of the absorber. Due to the interfacial multipoles between Cobalt filled MWCNTs and Polyurethane, the microwave absorption of Cobalt filled MWCNTs/Polyurethane composites is enhanced. It is evident from the results that these Cobalt filled MWCNTs composites studied as promising radar absorbing materials (Table 4).

Declarations Conflicts of interest The authors state that they have no conflicts of interest.

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