

Development of Cobalt coated MWCNTs/Polyurethane composite for microwave absorption

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Abstract. This research work describes the design and method of development of microwave absorber and was conducted for analysis of reflection loss performance with the magnetic modifications of Multi-Walled Carbon Nanotubes (MWCNTs). Cobalt coated Multi-Walled Carbon Nanotubes composites were prepared by three step methods. Composites were developed with varying weight percentage of Cobalt (II) Chloride Hexahydrate and Multi-Walled Carbon Nanotubes. The morphology, elementary analysis and absorbing properties of Cobalt coated Multi-Walled Carbon Nanotubes composites were studied by FESEM, EDX and Vector Network Analyzer. The obtained Co coated MWCNTs/PU composite demonstrated the maximum reflection loss of -21.06 dB at 12.63 GHz and the maximum absorption bandwidth of 3.7 GHz, in the frequency range of 8-13 GHz with 3 mm thickness. These microwave absorption parameters 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: co-precipitation method; electromagnetic; microwave absorption; Multi-Walled Carbon Nanotubes (MWCNTs); reflection loss

1. Introduction

Radar Absorbing Material (RAM) has a developing an extensive interest because of their various domestic and defense applications. The growth of RAM that projects in the microwave frequency had attained a great interest due to their prospective applications in the aerospace and defense stealth technology (Vinoy and Jha 1996, Lin *et al.* 2008, Ganesh *et al.* 2017). Rapid development in modern science and technology finds electromagnetic interference as a significant issue. There are many issues produced by the growing usage of electromagnetic waves (Singh *et al.* 2013, 2014, Saini and Choudhary 2013). So, it becomes essential to design and formulate commercial, light-weight and efficient radar absorbing materials to reduce electromagnetic interference, which have fascinated much attention of many researchers (Qing *et al.* 2009, Liu *et al.* 2008, Kaur *et al.* 2015a, b). Microwave absorbing materials are of two types: dielectric absorbing materials and magnetic absorbing materials. An ample search has been carried out for composite

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materials that possess the distinctive blend of electric, magnetic and dielectric properties suitable for reducing electromagnetic noise. Magnetic absorbers have exceptional absorptivity and high densities. Dielectric absorbers have low densities, but does not match the absorption capability of magnetic absorbers (Mathur *et al.* 2008). The Carbon Nanotubes (CNTs) i.e., dielectric material, are discovered in 1991 by Iijima. CNTs has stimulated intense curiosity in many research areas because of its anomalous electronic, mechanic, chemical and physical properties due to strong atomic bonding. Carbon nanotubes are categorized as, Single-Walled Carbon Nanotubes (SWCNTs) and Multi-Walled Carbon Nanotubes (MWCNTs). CNTs have high tensile strength which is much greater than that of steel which makes it a good candidate as a microwave absorber. Also, CNTs have high stiffness and an enormous aspect ratio which make them a prospective structural element for the enhancement of mechanical properties of microwave absorber composites (Mathur *et al.* 2014, Kumar *et al.* 2017, Wu *et al.* 2018). CNTs are filled with various kind of fillers such as polymers, metal nanoparticles, etc which enhances their electrical conductivity and thermal conductivity with low density (Zhao *et al.* 2010, Saini *et al.* 2011, 2012, Singh *et al.* 2012, 2014). The magnetic variation of CNTs makes them possess exceptional electromagnetic characteristics. The magnetic modified CNTs have prospective applications as microwave absorbing materials due to enhancement of both dielectric properties and magnetic properties. Different methods have been discovered for the fabrication of metal-carbon nanotubes compounds by filling metals into carbon nanotubes and by coating metals on carbon nanotubes via different techniques i.e., wet chemical method, co-precipitation method, electron-beam deposition, electroless plating etc, to combine the magnetic elements and CNTs, in which metals used as a magnetic material to modify CNTs (dielectric material) and enhance their microwave absorption performance (Tianjiao *et al.* 2011, Singh *et al.* 2011).

Various studies have been conducted to fabricate microwave absorber by magnetic modification of CNTs by filling or coating Zn, Fe, Ni and Co metals on it which have enhances microwave absorption by alteration of dielectric and magnetic characteristics. There are some analyses reported on MWCNTs with different composite materials which depicts the microwave absorption properties of the composites (Panwar and Lee 2019, Yusuf *et al.* 2020, Kumar *et al.* 2019, Iqbal and Ahmad 2020, Raveendran *et al.* 2019, Setua *et al.* 2020, Singh and Aul 2020). Porous Carbon and Cobalt nanocomposite which shows maximum reflection loss of -40 dB at 4.2 GHz with the thickness of 5 mm (Liu *et al.* 2008). Carbon Paraffin Wax composite with 50 wt% nanoparticles, exhibits the maximum reflection loss of -43.4 dB at a thickness of 2.3 mm (Liu *et al.* 2016). Carbon-encapsulated cobalt nanoparticles exhibit maximum reflection loss of -52dB with 13.26 GHz bandwidth in frequency range 2-18 GHz (Zhang *et al.* 2014). MWCNTs/Nickel and wax shows reflection loss of -20 dB, the matching thickness is 1.5 mm and an absorbing frequency band is 11.6–12.4 GHz (Deng and Han 2007). BaFe₁₂O₁₉/Ni_{0.5}Co_{0.5}Fe₂O₄/Carbon fiber nanocomposites were synthesized and shows the maximum reflection loss of -45 dB at 12.4 GHz with 2 mm thickness and 4 GHz bandwidth in 8-13 GHz frequency range (Zhu *et al.* 2020). Cobalt based Metal organic frameworks (70%), strontium hexaferrite (29%), and carbon nanofibers (1%) nanocomposite shows maximum reflection loss of -19 dB with 2.5 mm thickness and 3.9 GHz bandwidth (Zheng *et al.* 2020). Nitrogen doped cobalt/cobalt oxide/carbon/reduced graphene oxide nanocomposites (25%), shows maximum reflection loss of -63.0 dB with 1.66 mm thickness and 4 GHz bandwidth in 2-18 GHz frequency range (Shu *et al.* 2020a). Cobalt/Nitrogen doped CNTs had maximum reflection loss of -46 dB with 2.5 mm thickness and 7.2 GHz bandwidth in 2-18 GHz frequency range (Yan *et al.* 2019). Copper/Cobalt nickel ferrite/Graphene oxide/Polyaniline tri-composite was fabricated. When the concentration of tri-composite was 40%, it shows

maximum reflection loss of -33 dB at 10.8 GHz with 2 mm thickness in 2-18 GHz frequency range (Sun *et al.* 2020). NiCoMoO₄/CNTs shows the maximum reflection loss of 8.27 dB at 12.4 GHz with 2 mm thickness in 12-18 GHz frequency range (Bhardwaj *et al.* 2019). Co₃O₄/MWCNTs/Graphene composite had maximum reflection loss of -42.56 dB at 0.39 GHz with 5 mm thickness and 1.29 GHz bandwidth with 2 mm thickness in 1-3 GHz frequency range (Tao *et al.* 2019). The polyhedral Iron cobalt alloy/Graphite nanosheets binary composites were prepared. At 40 wt%, maximum reflection loss was -53.96 dB with 1.3 mm thickness and 4.3 GHz bandwidth in 2-18 GHz frequency range (Su *et al.* 2020). MWCNT/Zn_{0.25}Co_{0.75}Fe₂O₄/silicone rubber nanocomposite were fabricated which shows maximum reflection loss of -79.08 dB at 10.5 GHz with 2.4 mm thickness and 2.7 GHz bandwidth in X-band (Peymanfar *et al.* 2019). Nitrogen doped MWCNTs/Cobalt-zinc ferrite/Paraffin wax hybrid composites were synthesized. The minimum reflection loss was -64.7 dB with 3.1 mm thickness and 4.3 GHz bandwidth with 2.1 mm thickness in X-band (Shu *et al.* 2019a). It has been seen those magnetic modifications in CNTs greatly affects the microwave absorption characteristics by alteration of magnetic and dielectric properties of material.

This research is devoted to the fabrication of cobalt coated MWCNTs/PU composites for electromagnetic waves absorption. Main principle of this work was to coat the magnetic nanoparticles into the MWCNTs and to study material characteristics i.e., morphology & 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) Chloride Hexahydrate (CoCl₂.6H₂O), Sodium Hydroxide, 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%) to reflux for 7 h to remove trace impurities via water bath refluxing technique. Refluxed mixture was filtered with distilled water until pH 7. Further it was dried at 50°C for 12 h in hot air oven which results in functionalized MWCNTs



Fig. 1 Process of functionalization of MWCNTs

Table 1 Composites & its compositions

Sample code	Molarity (M)	Mass of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (gm)	MWCNTs (mg)
1	0.14	20.81	250
2	0.082	12.22	250
3	0.057	8.48	250
4	0.024	3.57	250

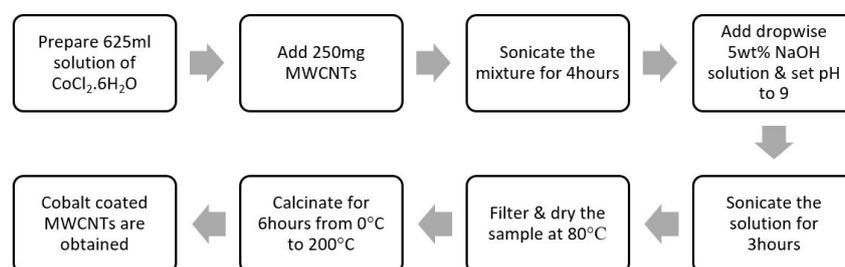


Fig. 2 Preparation of Cobalt coated MWCNTs

(Wang *et al.* 2010, Hao *et al.* 2010, Liu *et al.* 2009, Dong *et al.* 2009, Song *et al.* 2010, Rosca *et al.* 2005, Fan and Xin 2012, Mathur *et al.* 2016). The detailed experimental process of prepared sample is shown in Fig. 1.

2.2.2 Cobalt coated MWCNTs

Functionalized MWCNTs were coated with cobalt by the co-precipitation method with a simple thermal treatment process (Lang *et al.* 2011). Firstly, 625 ml solution of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ of different molar concentration as shown in Table 1, was sonicated. Then, 250 mg MWCNTs was added into $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ solution. Afterwards, the pH value of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ /MWCNTs solution was obtained upto to 9 by dropwise adding 5wt% NaOH at room temperature. Then, again solution was sonicated for 3 hours then, filtered and dried at 80°C in hot air oven. Then, the composites were calcinated, ramping from 0°C to 200°C for 6 hours in muffle furnace. The detailed composites composition and experimental process of prepared samples are shown in Table 1 and Fig. 2.

2.2.3 Preparation of Cobalt coated MWCNTs/PU composites

Cobalt coated MWCNTs were dispersed in N, N-Dimethylformamide (DMF) for 6hours using ultra sonication and Polyurethane (PU) were dissolved in DMF in another beaker using a magnetic stirring for 6 hours (Verma *et al.* 2017, Gupta *et al.* 2013a, b). The dispersed MWCNT and PU were mixed thoroughly using magnetic stirrer for next 6 hours. The solutions were dried in a vacuum oven at 3 kPa (22.5 mm of Hg) for next 6 hours 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 hours. The detailed weight percentage composition and experimental process of prepared samples are shown in Fig. 3 and Table 2.

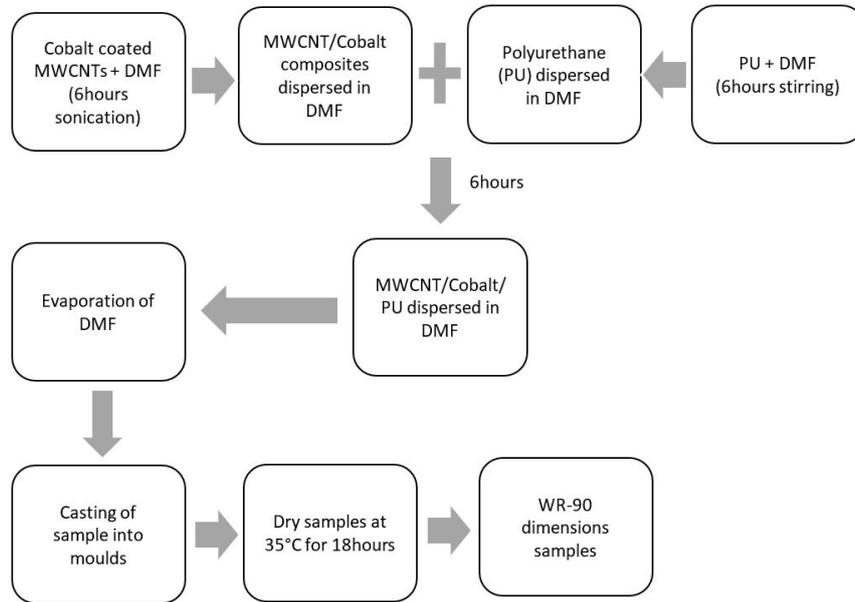


Fig. 3 Process of Composites: Cobalt coated MWCNTs/PU

Table 2 Weight percentage of composites

Sample code	CoCl ₂ .6H ₂ O (wt%)	MWCNT (wt%)	PU (wt%)
CMP 1	94.33	1.13	4.53
CMP 2	90.72	1.85	7.42
CMP 3	87.15	2.56	10.27
CMP 4	74.06	5.18	20.74

3. Results and discussion

3.1 Morphology and element analysis of Cobalt coated MWCNTs composites

Co-coated CNTs are obtained in the form of black voluminous powder. Field emission scanning electron microscopy (FESEM) was used to understand the morphology of the nano composites. The tubular MWCNTs have 5-20 nm diameter and 2-10 μm length, very cleanly, slippery, curved and twisted tubes. While functionalized MWCNTs have suspended bonds which were created the after acid treatment of MWCNTs. because of functionalization. During acid treatment, Carbon-Carbon bond breaks or the head caps of MWCNTs gets opened which led to increase the suspended bonds and functional groups on MWCNTs surface. These suspended bonds and functional gets replaced with further chemical treatment and Cobalt coats on it uniformly.

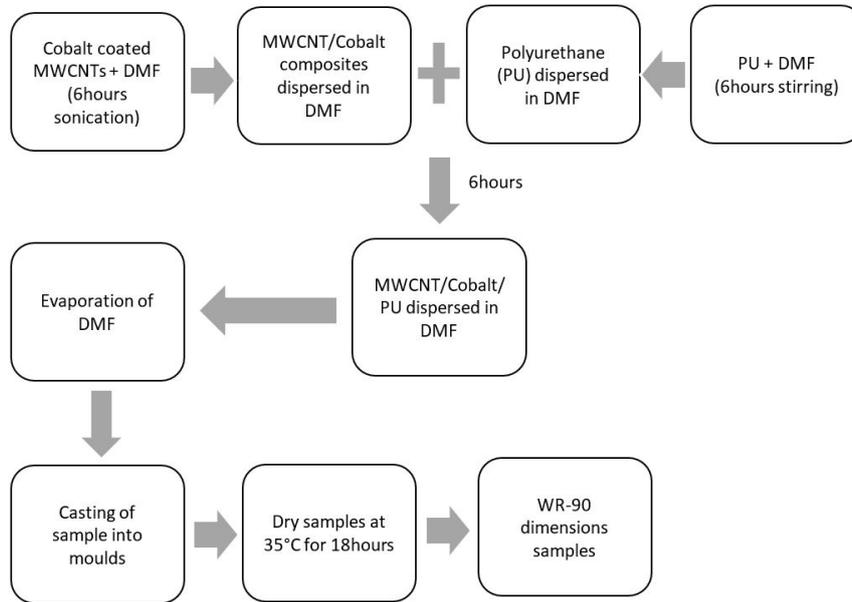


Fig. 3 Process of Composites: Cobalt coated MWCNTs/PU

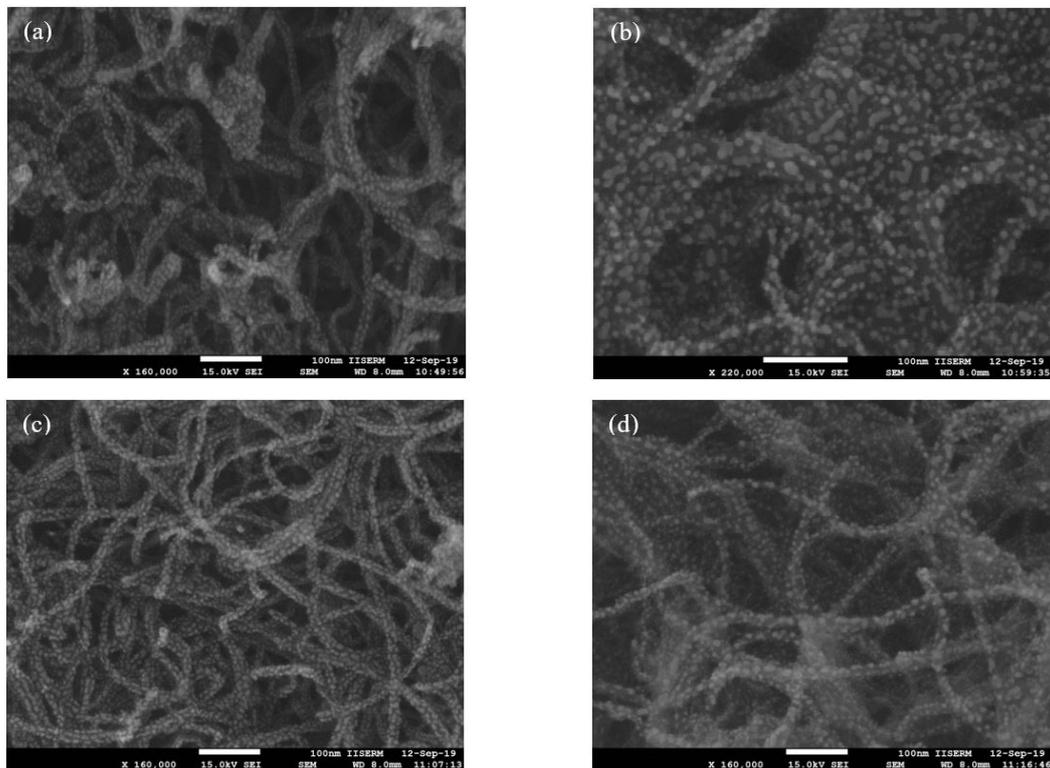


Fig. 4 FESEM of Cobalt coated MWCNTs with varying Cobalt composition (a) CMP 1; (b) CMP 2; (c) CMP 3; (d) CMP 4

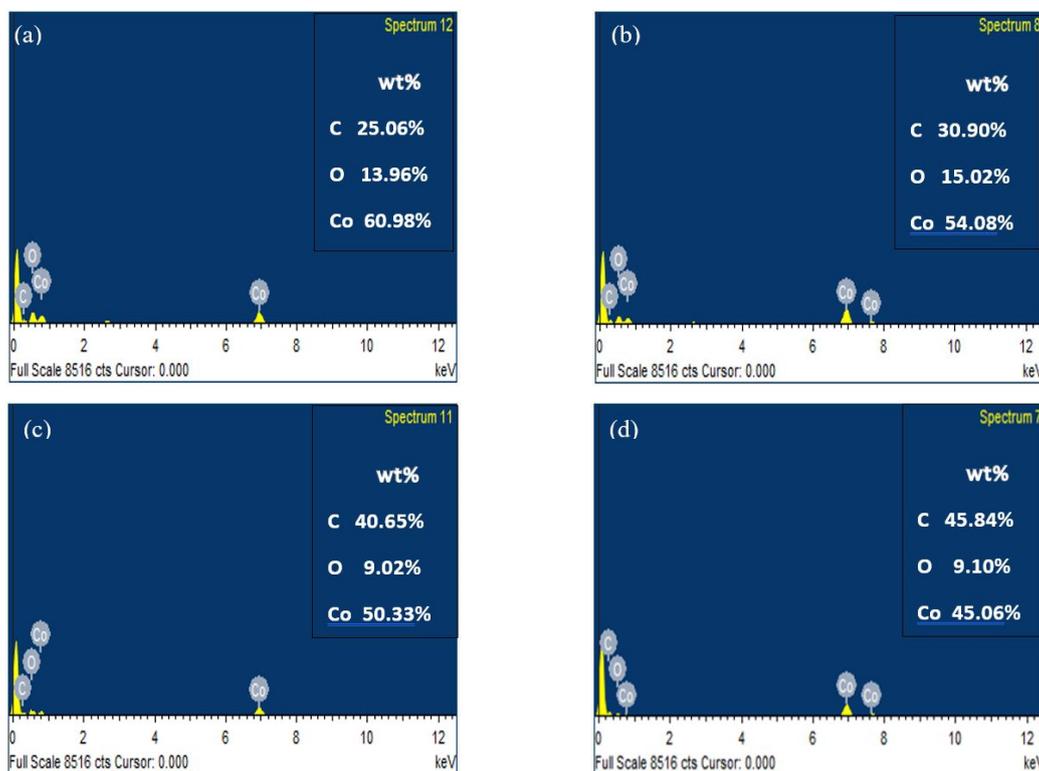


Fig. 5 EDX analysis of Cobalt coated MWCNTs with varying Cobalt composition (a) CMP 1; (b) CMP 2; (c) CMP 3; (d) CMP 4

CMP 1 has 0.14 M (20.81 gm) $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 250 mg MWCNTs. Fig. 4(a) shows the morphology of cobalt coated MWCNTs in which each MWCNT is coated homogeneously by cobalt nanoparticles. In this figure the approximated length of MWCNT is 169 nm and length of cobalt nanoparticle was 20-26 nm. CMP 2 has 0.082 M (12.22 gm) $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 250 mg MWCNTs. Fig. 4(b) shows the morphology of cobalt coated MWCNTs in which each nanotube is surrounded by cobalt nanoparticle. CMP 3 has 0.057 M (8.48 gm) $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 250 mg MWCNTs and CMP 4 has 0.024 M (3.57 gm) $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 250 mg MWCNTs. Figs. 4(c) and (d) shows the morphology of MWCNTs coated by cobalt nanoparticles. It is visible that as the concentration of cobalt decreases the MWCNTs becomes more visible which is shown in Fig. 4. Fig. 5 shows the EDX spectrum of Cobalt coated MWCNTs which represents the element analysis and weight percentage of element present in each sample of prepared nanocomposites. Fig. 5(a)-(d) shows the presence of carbon, oxygen and cobalt in each nanocomposite. Each nanocomposite has varying weight percentage of elements due to variation of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and MWCNTs.

3.2 Microwave reflection properties of Cobalt coated MWCNTs/PU composites

Microwave reflection properties of Cobalt coated MWCNTs/PU composites are characterized by Vector Network Analyzer (VNA). In this work, Keysight Technologies 20 GHz Vector Network Analyser is used to calculate the reflection loss of material. The sample is loaded in WR-90

waveguide in the shape which exactly fits inside it. In this method, two co-axial cables are used to calculate the reflection loss of Material Under Test (MUT). These are designed to calculate reflection loss in the range of 8 to 13 GHz. Before Measurement, Calibration of VNA is required. There are two steps for measurement of reflection loss. First step is known as Vector Error Correction (Calibration Phase). Vector-error correction is the process of characterizing systematic error terms by measuring known calibration standards, and then removing the effects of these errors from subsequent measurements. The method used for the calibration before measurement of reflection loss is called one port calibration method. For the calibration, the empty WR-90 Waveguide is inserted between the two port co-axial cables of VNA and then VNA is calibrated. Second step is known as Calculation phase. After the calibration is done, for calculation of reflection loss, the material under test is placed inside the WR-90 Waveguide and then it is placed between two ports which are further attached to vector network analyser. After then the calculation for the reflection loss of material performed by the VNA. The reflection loss performance is calculated by WR-90 dimensions [22.86 mm (length) \times 10.16 mm (width) \times 3 mm (thickness)] absorber. The reflection loss spectra in the range of 8-13 GHz is observed with same wt% MWCNTs and different cobalt concentration are summarized in Fig. 6. From the curves, it is observed that the maximum reflection loss is observed for CMP 4 is -21.06 dB at 12.63 GHz and minimum reflection loss is observed for CMP 2 is -3.40 dB at 9.48 GHz. CMP 4 has maximum reflection loss of -21.06 dB at 12.63 GHz frequency with 3.7GHz bandwidth window. CMP 4 has $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (74.06%), MWCNTs (5.18%) and PU (20.74%) composition. The overall maximum reflection loss and bandwidth corresponds to magnetic and dielectric compositions of prepared sample. CMP 4 has least amount of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ which provides magnetic as well as dielectric properties of material. While all other samples have the larger amount of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ composition as compared to MWCNTs composition which may suppresses the dielectric properties in comparison to magnetic properties. It has been observed that the maximum bandwidth window is available for CMP 3 i.e., 4.53 GHz in the frequency range of 8-13 GHz shown in Table 2. CMP 3

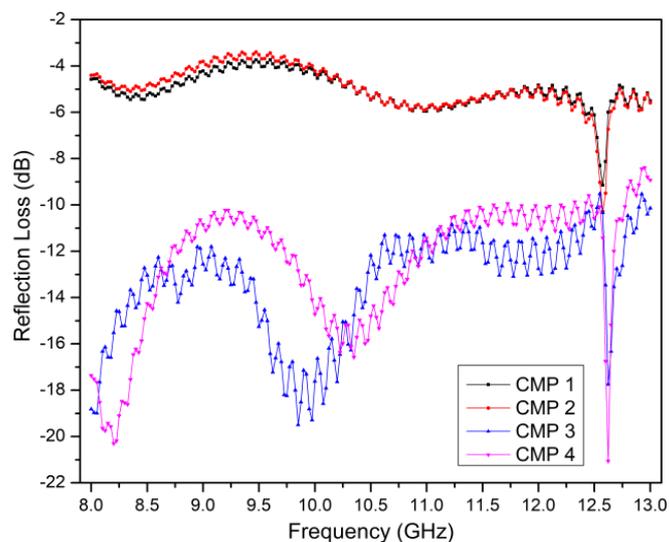


Fig. 6 Microwave absorption characteristics of the composites with varying compositions of Co/MWCNTs/PU

Table 3 Analysis of prepared composites absorber in term of Thickness, Reflection Loss 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)
CMP 1	3	-9.15	12.58	-
CMP 2	3	-10.28	12.58	-
CMP 3	3	-19.50	9.85	4.53
CMP 4	3	-21.06	12.63	3.7

Table 4 Microwave absorption characteristics comparison with previously reported and present work

Material (wt%)	Matrix	Thickness of absorber (mm)	Maximum reflection loss (dB)	Frequency for maximum reflection loss (GHz)	Bandwidth (GHz)	Reference
Carbon black (50%)	Polyethylene	2.4	-17.5	10.1	0.6	Ansari and Akhtar (2018)
MWCNTs (2%)	Epoxy resin	2.24	-25	10.9	3	Kim <i>et al.</i> (2008)
Co/CNTs (20%)	Epoxy resin	1.8	-20	8	2.8	Wu <i>et al.</i> (2016)
Co-C/MWCNTs (25%)	Paraffin	2.5	-28	10	3.6	Shu <i>et al.</i> (2019b)
Co/C/MWCNTs (15%)	Paraffin	2.55	-48.9	8.2	2.3	Yin <i>et al.</i> (2017)
Co/NC/MWCNTs (15%)	ZIF-L	1.5	-44.3	5.20	1.7	Xu <i>et al.</i> (2019)
MWCNT/ Zn _{0.25} Co _{0.75} Fe ₂ O ₄	Silicone Rubber	2.4	-79.08	10.5	2.7	Peymanfar <i>et al.</i> (2019)
MWCNT/ Zn _{0.5} Co _{0.5} Fe ₂ O ₄	Paraffin Wax	3.1	-64.7	8	2	Shu <i>et al.</i> (2019a)
MWCNTs (2.56%), CoCl ₂ .6H ₂ O (87.15%)	Polyurethane	3	-19.50	9.85	4.53	This work
MWCNTs (5.18%), CoCl ₂ .6H ₂ O (74.06%)	Polyurethane	3	-21.06	12.63	3.7	This work

has CoCl₂.6H₂O (87.15%), MWCNTs (2.56%) and PU (10.27%) composition. So, the maximum window occurs due to enhanced magnetic properties over dielectric properties. Maximum reflection loss and its bandwidth window for each sample is shown in Table 3. The obtained nanocomposites exhibited excellent comprehensive microwave absorption properties with strong

absorption, broad bandwidth and thin thickness. It was noteworthy that the additive amounts of Co, MWCNTs and filler loadings should be carefully regulated for obtaining optimal microwave absorption performance. Microwave absorption characteristics of previously reported and present work are shown in Table 4 and present study achieves the broad bandwidth characteristics. Therefore, this work could be valuable for developing carbon-based magnetic composites as lightweight and high-efficiency microwave absorbers.

The possible microwave absorption mechanisms of Cobalt coated MWCNTs/PU could be credited to the following characteristics. Firstly, the residual oxygen containing functional groups such as COOH and OH, and structure defects on the surface of MWCNTs could induce the dipole polarization and defect polarization under the alternating electromagnetic fields. Secondly, numerous heterogeneous interfaces among polyurethane matrix, MWCNTs and Cobalt significantly enhance the interfacial polarization relaxation. Thirdly, Co coating on the crystal lattice of MWCNTs, which not only enhances the dipole polarization relaxation, but also improves the conduction loss. Fourthly, according to the Cao's electrons hopping model, the capacitor-like structure at the interfaces could attenuate the power of incident microwaves by aligning the polar bonds or charges under the alternating electromagnetic fields. Thus, the electrons can absorb electromagnetic energies to migrate on the surface of MWCNTs, and then convert the electromagnetic energies into thermal energies by colliding with the lattice. Lastly, the synergistic effects of dielectric loss derived from interfacial polarization, defect polarization and dipole polarization, magnetic loss originated from ferromagnetic Co nanoparticles, and conduction loss coming from MWCNTs, which significantly improve the microwave attenuation capacity and optimize the impedance matching (Deng *et al.* 2018, Feng *et al.* 2017, Shu *et al.* 2018, Lv *et al.* 2016a, b, 2017a, b, Shu *et al.* 2019c, 2020b, c, Qiao *et al.* 2020, 2021).

4. Conclusions

In this work, Cobalt coated MWCNTs/Polyurethane composites were fabricated. The characterizations of Cobalt coated MWCNTs/Polyurethane composites were investigated by FESEM, EDX and VNA in the frequency range of 8-13 GHz. The maximum reflection loss achieved was -21.06 dB at 12.63 GHz with 3 mm thickness and 3.7 GHz bandwidth at Cobalt (II) Chloride Hexahydrate (74.06%), MWCNTs (5.18%) and PU (20.74%) composition. The maximum bandwidth was 4.53 GHz with 3mm thickness at Cobalt (II) Chloride Hexahydrate (87.15%), MWCNTs (2.56%) and PU (10.27%) composition. The reflection loss enhancement of the Co coated 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 coated MWCNTs and Polyurethane, the microwave absorption of Cobalt coated MWCNTs/Polyurethane composites is enhanced. Therefore, the obtained Cobalt coated MWCNTs composites demonstrated potential applications in the fields of electromagnetic absorption.

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