International Journal of Engineering &Technology, 8 (2) (2019) 102-109

International Journal of Engineering & Technology

Website: www.sciencepubco.com/index.php/IJET

Research paper

Absorption properties of TiO2 coated MWCNT/FE3O4/poly aniline Nano composite in x-band

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Abstract

The aim of current research is construction and investigation of radar absorbing material (RAM) -TiO2@MWCNT/Fe3O4/PANI nano composite. In order to construction of nano composite sample; we prepare 20% weight of TiO2@MWCNT nano fillers (by sol gel method) , Fe3O4 and PANI in separate form and then we used proper solution in ultrasonic bath by adding epoxy resin and hardener with nano composite solution blending method in X band standard size (22.8*10.16 mm2) and 3mm thickness. Phase and morphology features were investigated by X ray diffraction analysis and Scanning electron microscopy. RAM- TiO2@MWCNT/Fe3O4/PANI nano composite mi-crowave features were studied at X band at the range of 8-12 GHz by vector network analyzer. Reflection loss has two minimum peak such as -28.59 dB at 9.36 GHz and -33.77 dB at 10.9 GHz. Electromagnetic parameters such as permeability, permittivity, tangent loss and refractive coefficient were studied. Results showed that using proper nano fillers to combining dielectric and magnetic losses microwave absorption increasing.

Keywords: *Fe3O4; Permeability; Permittivity; TiO2@MWCNT; X-band.*

1. Introduction

Fast development of electronics systems and using electromagnetic waves cause to creating problems in military and communication affairs specially in microwave region. So it is inevitable using of waves absorber to electronics systems conservation against electromagnetic waves disturbed, war facilities camouflage in enemy radar view and maintaining possibility risks in body against waves absorption [1]. We demand to use light, flexible microwave materials in commercial applications [2]. For example, we can refer to cellular system, wireless LAN devices and wireless antenna system in computer [3]. Microwave absorbers because of features such as strong absorption, very high frequency range, low thickness and low density have vital and important roles. If particles size reduces to nano scale; microwave absorber will be high to effect of quantum size [4]. So edge width increase by lowering of particles size. Also coupling interaction increase in edge width. Higher surface area increases in finer particles and this case cause to creating more dipoles and dipole polarization improve microwave absorber features [5].
Main point in absorber microwaves designing relate to optimum adjusting electromagnetic, electrical [6] and thickness percent [3]. Most of magnetic fillers combine with metal in high density such as hexagonal ferrites and metallic magnetic materials [7]. Fe_3O_4 is a cheap, stable material in high temperatures, low toxic percent, reversible spinal structure, high amount of magnetic saturation, high magnetic crystals anisotropy constant and high electrical resistance is a best and proper choice for absorber microwave in GHz range or spectrum [8]. In order to observing complex high percent absorption, we need high weight. Against of magnetic fillers, MWCNTs are as dielectric absorber with low density proportion and low complex ratio. Many composites were reported in current field based on CNTs [9]. They have excellent electrical conduction, well thermal stability, resistance against chemical agents and high ratio. Also CNTs dispersion power in composite matrix help to improving function [10]. Polymeric matrix, electrical and mechanical function increase with little percent. Dioxide titanium has acceptable features such as 100 average dielectric constant, high thermal stability and energy conversion. Specially nano Dioxide titanium because of small size, surface, quantum size effect on microwave features and it improve mentioned cases [11]. Conductor polymer and inorganic magnetic nano particles combining have conduction and magnetic features. So, interest to designing and materials construction controlling were developed by conduction and magnetic features specially with poly aniline. Poly aniline [12] as an excellent conductor polymer.
It has many applications because of monomer, simple construction method, high chemical and thermal stability, low special mass and high conduction in microwave frequencies. According to mentioned details, nano composites with magnetic and dielectric materials and optimum conduction is the best choice for microwave absorber [13]. It has combination two cases loss. In current research, we reached to proper results about microwave features in X band by constructing RAM- TiO_2@ MWCNT/ Fe_3O_4/ PANI nano composites and increasing fillers number against previous works. Nano composite with 20% weight percent includes 3 fillers such as TiO_2@MWCNT, Fe_3O_4 an PANI and they were dispersed in resin epoxy [14] matrix and also nano composite was prepared with 3mm thickness.

2. Theory

Radar absorbing material mechanism depend on proper impedance matching among dielectrics and magnetic materials. Some materials combination with different particles size and shape relate to multiple dispersion and arouse electromagnetic energy loss in thermal form [15]. Surface effect, elements anisotropy and asymmetry distribution and porosities play an important role in loss mechanism. Dipole polarization and ionic conduction interactions are another mechanism which they effect on radar absorber. Absorber materials function in electromagnetic fields determine by free electron displacement and their bounding in electrical fields, atomic movement direction in magnetic fields which they contain magnetic losses (eddy current losses, magnetic remained a waste losses) [16] and electrical losses (conductance, dielectric interaction and severe losses). Geometrical and 0.25 wavelength effects are main points for strong absorber. Effective factors in electromagnetic waves absorber determine by permittivity, permeability and electrical conducting. Reflective loss less than -10 dB means 90% absorption performance.

$$RL = 20\log\left(\frac{Z_{in} - Z_0}{Z_{in} + Z_0}\right) \quad (1)$$

Z_0 is free space impedance in ideal conditions for reflective loss with $Z_{in}=377\Omega$. In order to achieving mentioned aim; material Z_{in} certain impedance must be adjusting relative permittivity constant, relative permeability constant, frequency and absorber thickness and free space inside impedance which it is equal with $Z_{in}=377\Omega$ [17].

$$Z_{in} = Z_0\sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left(j\left(\frac{2\pi fd}{c}\right)\sqrt{\mu_r\varepsilon_r}\right) \quad (2)$$

3. Experiment

3.1. Agent MWCNT preparing

At first, we blend nitric acid and sulfuric acid (HNO_3, H_2SO_4) with 2:3 ratios [18], then we set string heater instrument at 50’C. Acid mixture, MWCNT and magnetite add to pot. They remain in certain state for 24 hours. They are washed with deionized water and they are dried at 100’C.

3.2. TiO2@MWCNT preparing

Agent MWCNTs, titanium (IV) n-butoxide, iso-propanol, aqua pura were applied with 1:0.3:50:25 weight ratios [19]. After titanium (IV) n-butoxide adding, sonic implement during 15 minutes. Aqua pura add to mixture slightly and we shake it. Mixture was located in constant case by stirrer at room temperature for 48 hours. Iso-propanol add to solution by heating at 100’C. Powder is located at furnace at 500’C during 100 minutes.

3.3. Fe3O4 preparing

$FeCl_2$ and $FeCl_3$ salts pour on 3 opening pot with 1:2 molar ratios and aqua pura with 100cc degas by N_2 in reaction region. In another opening, separated funnel with 10cc 25% ammonia are pour in system [20]. Degasification from aqua pura continue 20 minutes. Then weighted salts are poured in pot and they spin by magnetite and high rotation speed in solution. Reaction temperature is set at 80’C. When reaction temperature reach to 80’C, we will open separating funnel tap and all of ammonia is added to solution. Reaction continue to 45 minutes and tools are separated with each other. Fe_3O_4 particles are separated by strong magnet and they are washed with aqua pura in some stages. Finally, separated particles are located in oven and they are dried. They are rubbed in oven to change black powder.

3.4. Poly aniline (PANI) preparing

At first, 25 m L of distilled and fresh aniline is added to 500 m L acid chloric 2 molars in pot and they are put in ice batch. 250 m L ammonia persulfate 2 molars are stirred in aniline acid solution by drops [21]. By increasing oxidation in this stage; begins polymerization reaction. We give fortunate during 1 hours to reaction to change remained monomers in polymer in presence of oxidant. Then polymeric precipitation is washed with clear funnel and 500 m L of chloric acid 1 molar is washed twice stages to non-react of monomers from polymers. Polymer is washed at room temperature during 48 hours and it is dried in oven with 60-70’C.

3.5. Nano composite preparing

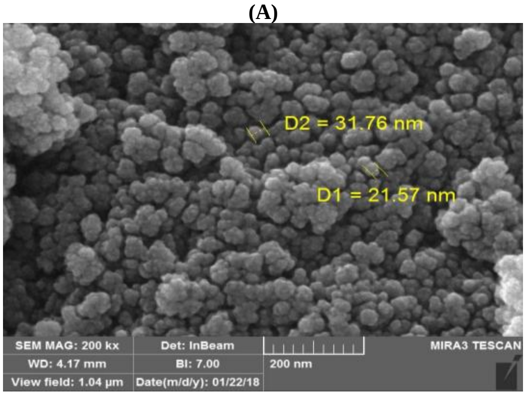
Many traditional methods were presented to fillers dispersion at matrix such as molten mixture, solvent process by helping eccentrics force, ultrasonic and active factors behavior on surface which solution blending is very important [22]. To nano composite construction with 20% weight, we have been used Fe_3O_4 magnetite, PANI poly aniline and TiO_2@MWCNT fillers equally for observing dielectric and magnetic loss combination. Epoxy resin was regarded as matrix to nano composite construction [23]. At first, TiO_2@MWCNT add to ethanol solution at ultrasonic batch, 60’C and it put on sonic during 30 minutes. Fe_3O_4 add to solution and it sonic 15 min and then PANI add to system and it sonic and finally epoxy resin add and it sonic 60 min. at 60’C and 18-hour solution dispersion was done. Hardener add to system after solution dispersion in proper frame during 18 hours with X waveguide by 22.8*10.16 mm^2 opening and 3mm thickness to microwave feature analysis. Sample is put on normal conditions during 48 hours to processing.

4. Result and discussion

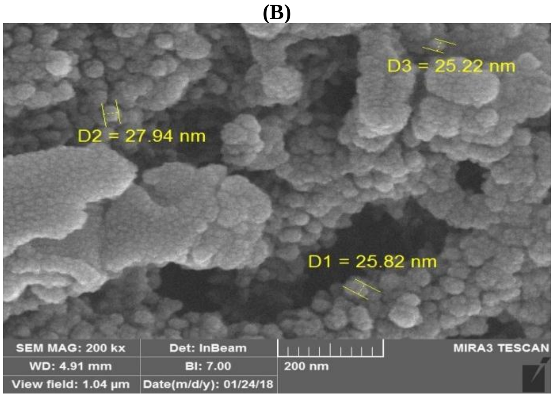
Figure 1 show SEM case of Fe_3O_4, PANI, MWCNT and TiO_2@MWCN particles. Figure 1a show SEM image of Fe_3O_4. Fe_3O_4 particles size are equal with 20-35 nm with circular surface morphology. Some of small balls were shown in nano ferrites and small particles size are effect on magnetic resonance which they cause to better behavior in GHz range. Figure 1b show SEM image of PANI particles. PANI particles size are equal with 25-30nm and its empty circles surface are shapeless, but generally they are monotonic. PANI empty circles are very soft without impurity. Figure 1c shows MWCNT particles. Particles size are between 20-30nm. Every particle size and anisotropy size play an important role in various anisotropy fields creating inside particles. Anisotropy fields increase absorption frequency bandwidth. Figure 1d shows SEM images of TiO_2@MWCNT particles. Particles size are between 22-32nm and surface morphology has TiO_2 monotonic on MWCNT. Particles size have specific effect on waves loss. Surface area increasing, atoms surface increasing and multiple reflection cause to increasing dielectric and magnetic losses and also they cause to more absorption of electromagnetic waves.

X-Ray diffraction (XRD) method: It is an important and direct method to determine phases type and material crystal structures. Figure 2 shows XRD for Fe_3O_4, PANI, MWCNT and TiO_2@MWCNT particles. Figure 2a shows XRD for Fe_3O_4 nano particles. This figure shows Fe_3O_4 in 2θ ,18.92, 30.28, 35.59, 36.6, 43.2 ,53.74 ,57.18 ,62.82, 73.99 and 74.5 amounts which they are (111), (220), (311), (222), (400), (422), (511), (440), (620) and (533) in crystal plates and square magnetic sample. Figure 2b shows XRD for PANI nano particles with peaks amounts extensively. They were observed in 2θ = 35.64, 33.75, 25.39 ,19.79 and 16.37. Figure 2c shows XRD for MWCNT with 2θ = 25.69, 43.55 amounts and (002), (101) crystal plates. Figure 2d shows XRD for TiO_2@MWCNT in 2θ = 25.38, 37.92, 48.10, 54.08, 55.14, 62.7, 67.10, 70.36 and 75.14 amounts (101), (004), (200), (105), (201), (204), (116), (220), (215) crystal plates. For TiO_2@MWCNT, multiple peaks were created against TiO_2 standard state with octahedral form. Severe peaks by creating in 25.38 are equal with (101) and XRD sample is confirmed by previous reports [24]. Coordinated peaks with octahedral form confirm TiO_2 generation during Sol gel coating process. Nano crystals size in nanometres surface area range, atoms dangling bounds number, unsaturated coordination increase on surface.

(A)



(B)



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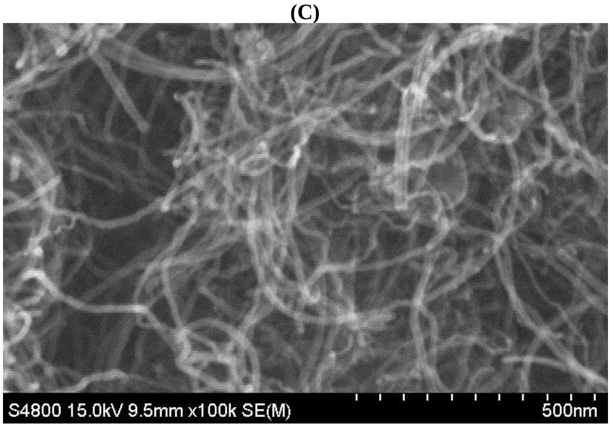

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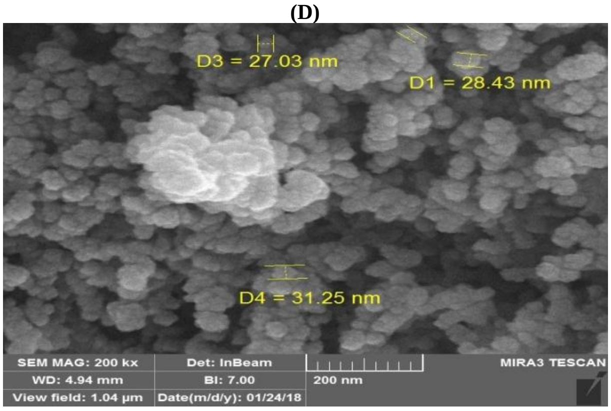
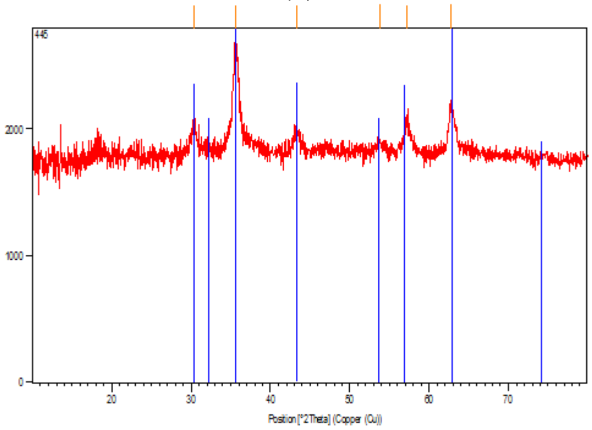


Fig. 1: SEM Images of (A) Fe3O4, (B) PANI, (C) MWCNT and (D) TiO2 Coated MWCNT.

(A)



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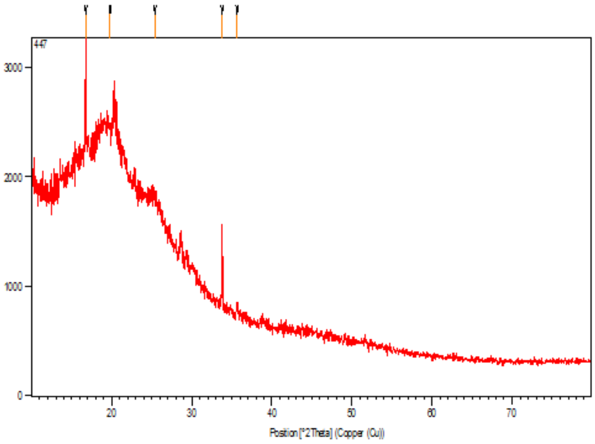
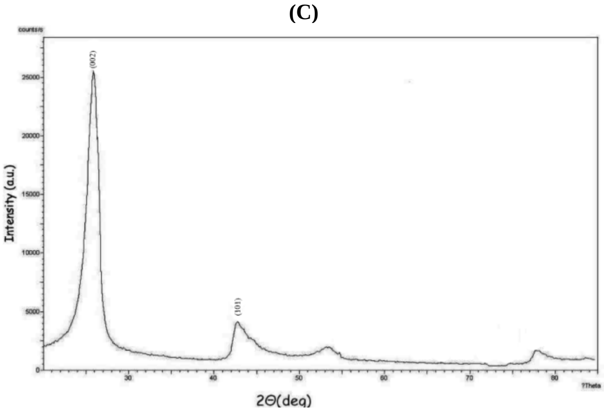


(C)



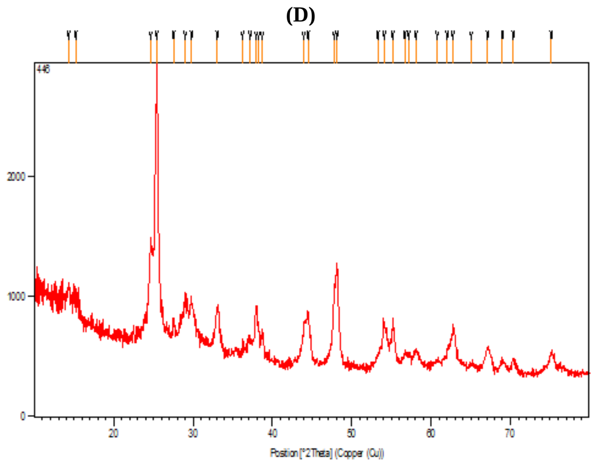


Fig. 1: XRD Analysis of (A) Fe_3O_4, (B) PANI, (C) MWCNT and (D) TiO_2 Coated MWCNT.

In current process microwave absorber features was calibrated by vector network analyzer (Agilent E8363C) instrument and then they were put on waveguide place with X band standard size (22.8*10.1 mm^2) and 3mm thickness, as shown in figure 3.

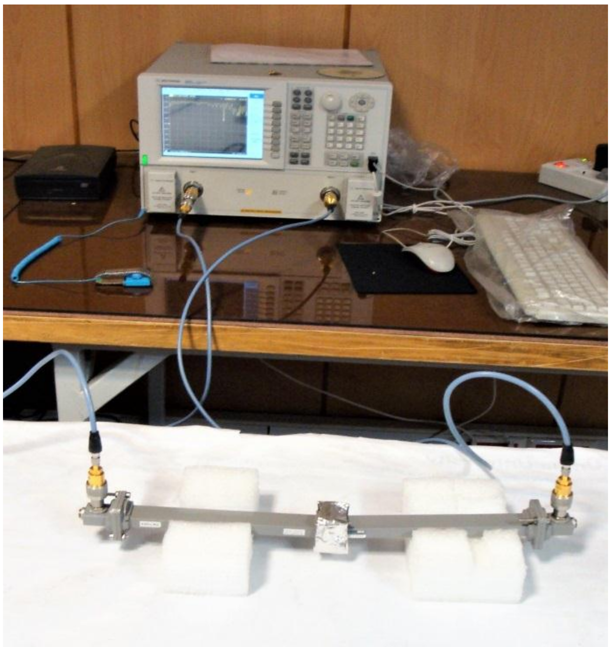


Fig. 3: Measurement System, Vector Network Analyzer.

After electromagnetic waves flashing in 8 to 12 GHz range in sample; S_{11}, S_{12}, S_{21} and S_{22} diffraction parameters were extracted from instrument. Middle spectrum and multiple dispersion are suitable for microwave more absorption. Based on retrieval technique [25], we computed complex permeability and permittivity constants. Complex permeability and permittivity constants amount were shown in figure 4 at 8 to 12 GHz for TiO_2@MWCNT/Fe_3O_4/PANI. In figure 4a; permeability (μ) was shown with imaginary (μ′) and real parts (μ″) and according to magnetic loss tangent formulae (4);

$$\mu = \mu' - i\,\mu'' \qquad (3)$$

$$\tan\delta_{\mu} = \frac{\mu''}{\mu'} \qquad (4)$$

Imaginary part is more than real part by regarding electromagnetic waves absorption. Fe_3O_4 particles cause to hysteresis and eddy current losses and magnetic loss. In figure 4b, permittivity constant real part (ε′) presents complex polarized power at external field effect and also permittivity constant imaginary part (ε″) named dielectric loss and it relates to electromagnetic energy conversion to heat or electromagnetic waves absorption by material.

$$\varepsilon = \varepsilon' - i\,\varepsilon'' \qquad (5)$$

$$\tan\delta_{\varepsilon} = \frac{\varepsilon''}{\varepsilon'} \qquad (6)$$

According to dielectric loss tangent equation (6), imaginary dielectric part is more than real part by regarding electromagnetic waves absorption which it clears well at 4b figure too. Many polarization mechanisms help to materials dielectric function which they are electronic, ionic, space charge polarization directed. In microwave frequency range, we can only use directing and space charge polarization. Space charge polarization is created from material heterogeneity and directed polarization in presence of bound dipole charges. Two mentioned polarizations with directing and space charge pattern play important roles in dielectric function. Figure 5a shows TiO_2@MWCNT/Fe_3O_4/PANI nano composite refraction coefficient and its negative amount investigate based on absorber material principals. Figure 5b shows TiO_2@MWCNT/Fe_3O_4/PANI nano composite dielectric losses tangent.

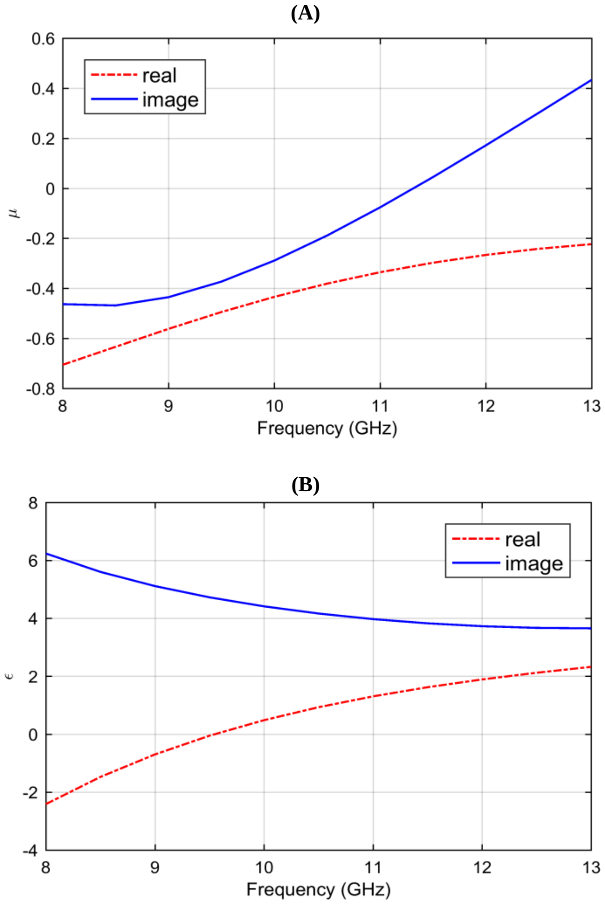
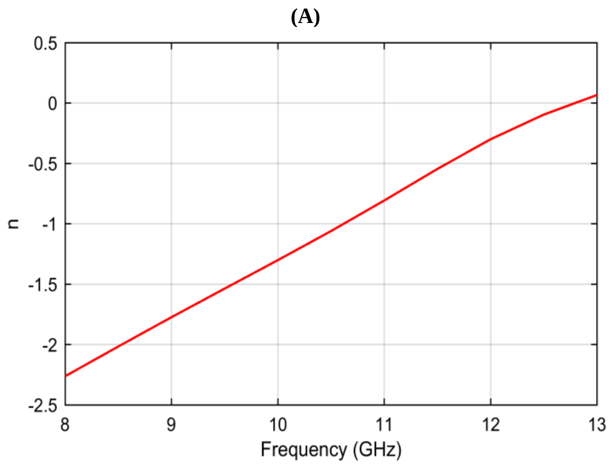


Fig. 4: (A) Complex Permeability and (B) Complex Permittivity of TiO2@MWCNT/Fe3O4/PANI-Epoxy Absorbers.

To computing conduction, we use main part of conduction in PANI. Microwave conductivity (σ_T) can be related to imaginary permittivity [26]:

$$\sigma_T = (\sigma_{ac} + \sigma_{dc}) = \omega \varepsilon_0 \varepsilon'' \quad (7)$$



(B)

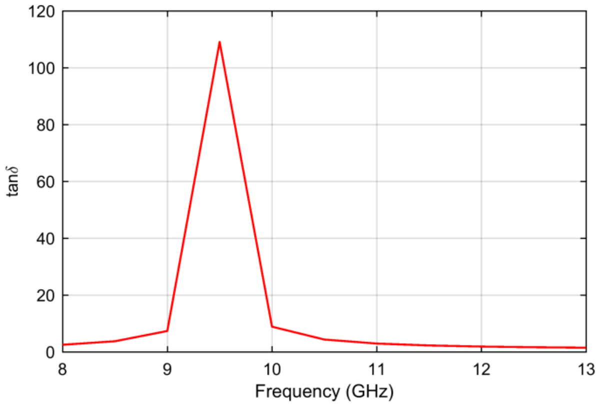


Fig. 5: (A) Refraction Coefficient and (B) dielectric Loss Tangent for the TiO_2@MWCNT/Fe_3O_4/PANI-Epoxy Absorbers

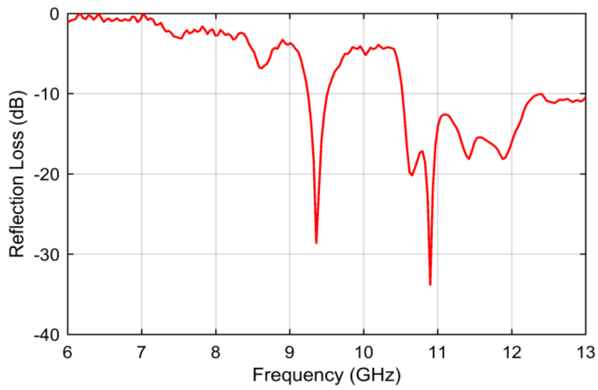


Fig. 6: Reflection Loss of RAM-TiO2@MWCNT/Fe3O4/PANI.

Where σ_{ac} and σ_{dc} are frequency dependent (ac) and independent (dc) components of σ_T respectively, ω is angular frequency and ε_0 is permittivity of free space [27]. Conduction increasing cause to eddy current and conductance losses. Of course material impedance with high conductance is less than air impedance. Therefore, this material has very small covering depth and electromagnetic wave reflect by material. In order to achieving the best absorption effect, it is necessary to use proper conductance in absorber material. Reflective loss is computed based on transmission line theory [28]. Figure 6 shows RAM- TiO_2@MWCNT/Fe_3O_4/PANI reflection loss. It has two minimum peak amounts which they are -28.59 dB at 9.36 GHz; and -33.77 dB at 10.9 GHz. Frequency band width in mentioned two parts is less than -10 dB. First part relates to 0.3GHz at 9.2-9.5 GHz; and the main part is happened 2.5GHz at 10.5-13 GHz. The relationship between electromagnetic reflectivity loss correlated with the percentage of absorbed energy is given in Table 1 [29]. Comparison of EM composite with another composites show similarities between constructed nano composite microwave absorber sample in investigated pattern and we reached relative result. Fillers percent, fillers type, construction method, composite matrix type, fillers number and absorber material thickness are as effective factors for results changing.

Table 1: Relation between Reflection Loss and the Absorbed Energy [29]

Reflectivity reduction, dB	Absorbed energy %
0	0
-3	50
-10	90
-15	96.9
-20	99
-30	99.9
-40	99.99

Table 2: Comparison of RAM-TiO2@MWCNT/Fe3O4/PANI with another Composites

References	Reflection loss (dB)	Freq.(GHz)	Thickness (mm)	Radar Absorber Material (RAM)
[20]	-15.33	5.4	6	Fe_3o_4
[20]	-28.38	5.4	6	Ni-B/fe_3o_4
[11]	-26	6.5	3	Tio_2@MWCNT
[11]	-10	12	3	MWCNT
[11]	-4.7	10.14	3	Tio_2
[30]	-16	10.99	2	MWCNT(%30)
[30]	-8.4	12.4	2	Ti(%30)
[30]	-36.54	12.05	2	Ti@MWCNT (%30)
[30]	-42.53	10.98	2	Ti@MWCNT/ Fe(%30)
[12]	-0.75	11	2	PANI
[12]	-5.3	9.2	2	Fe_3o_4-PE@PANI
[10]	-21.1	11.5	5	BNCNT
[10]	-39.5	13.3	3.5	BNCNTs/Fe_3O_4
[10]	-51.2	11.7	5	BNCNTs/Fe_3O_4
This Work	-33.77	10.9	3	TiO_2@MWCNT/ Fe_3O_4/ PANI(%20)

5. Conclusion

TiO_2@MWCNT, PANI and Fe_3O_4 fillers were synthesized successfully and RAM- TiO_2@MWCNT/Fe_3O_4/PANI nano composite was prepared with 3mm thickness and 20% weight. The materials were characterized by XRD and SEM. Microwave absorber behavior was investigated by vector network analyzer. The Minimum reflection loss was obtained in -28.59 dB at 9.36 GHz and -33.77 dB at 10.9 GHz amounts which they are combination of dielectric and magnetic losses. Dielectric loss was gave from TiO_2 and MWCNT, and magnetic loss was gave from Fe_3O_4. We have been used PANI high conductance and also we regarded features such as quantum size, middle polarization, multiple dispersion, coupling interaction, dipole polarization which they cause to microwave absorber characteristics.

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