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Study of Reflection Loss In Ku Band By CNM Decorated With Metal Nano Particles

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Abstract: ***The study of microwave absorption and thus reflection loss in Ku band has received much attention recent years. 12 – 18 GHz microwave frequency band mainly used for tracking of military objects through radar i.e stealth technology of aircraft anti radar detection and wireless, satellite communication applications. Carbon nanomaterials (80-90 nm) decorated with metal nanomaterials (40-50 nm) were synthesized from plant-based precursors using Taguchi optimization methodology. The samples under study showed reflection loss to the extent of -37.11 dB at 14.31 GHz and -34.78 dB at 17.76 GHz whereas, -5 dB RL was observed throughout the Ku band frequency range and importantly in most cases the samples showed -10 dB loss for 1 GHz bandwidth in the 14-15 GHz and 17-18 GHz frequency range. Thus, the synthesized materials could be a good microwave absorbing candidate for particularly 14-15 GHz and 17-18 GHz microwave frequency range bandwidth.***
Keywords: carbon nano materials, metal nano particles, microwave absorption, plant fibre, reflection loss.

I. INTRODUCTION

The study of microwave absorption (MA) and thus reflection loss in Ku band has received much attention recent years. 12 – 18 GHz microwave frequency band mainly used for tracking of military objects through radar i.e stealth technology of aircraft anti radar detection and wireless, satellite communication applications [1-2]. Carbon based microwave absorbing material have gained significant popularity because of their light weight, low density, accordingly, studies has been carried out extensively to developed new microwave absorbing materials with high dielectric loss and magnetic loss [3-5]. Further, researchers focused on nanostructure materials like nano ferrites, nano metals, nano composites and nano carbonaceous materials compare to microstructures materials to enhanced MA property [6-11]. Carbon nano materials specially designed to exhibit strong MA property over a wide frequency range with fascinating physical, chemical and mechanical property [12-13]. Therefore, nanomaterials have generated tremendous amount of research interest due to its intrinsic properties which is not observed in its bulk or micro counterpart's materials.

Many researchers have studied MA property in Ku band using nanomaterials and nanocomposites synthesized from non-renewable resources precursor for example S.H Hosseini *et. al* prepared nano composite of MWCNT/Ba0.2Sr0.2La0.6MnO3 and studied its MA property in 12 – 18 GHz and observed maximum reflection loss of -22.36 dB at 14 .78 GHz with a bandwidth of 2.67 GHz (more than -10 dB) [14]. Whereas Qing-Qing Ni *et. al* fabricate one dimensional CNT@Ba_3TiO_3@PANI heterostructure composite and investigated -28.9 dB reflection loss at 10.7 GHz with 3 mm thickness and less than -20 dB reflection loss had achieved from 10- 15 GHz. [15]. B. P. Singh *et. al* prepared single walled CNT attached with cobalt and nickel nanoparticles by dc- arc discharge technique using a high-density graphite block and observed its microwave shielding effectiveness value of 24 dB (blocking >99% radiation) in the frequency range of 12-18 GHz with 1.5 mm sample thickness [16]. Lokesh Saini *et. al* synthesis carbon coated nickel metal core nanoparticles using thermal treatment of Ni hydroxide in aniline-formaldehyde copolymer matrix under N_2 atmosphere and observed MA in Ku band at lower thickness of 1mm [17]. K.K. Gupta *et. al* coated the cotton fabric with Ni-Zn ferrites and evaluate the MA, transition, reflection and reflection loss in X and Ku band. Observed study shows 40% absorption, 20 % transmission, and 40 % reflectance in 8- 18 GHz frequency range [18]. Zetao Zhu *et. al* produced reduced graphene oxide – nickel composite, studied its MA property in Ku band and observed -42 dB refection loss at 17.6 GHz with 2 mm thickness [19]. Reza Peymanfar *et. al* prepared a magnetic $SrAl_{1.3}Fe_{10.7}O_{19}$ nanoparticles and multiwalled carbon nano tubes composites by sol-gel method and studies its MA property in X and Ku band. They reported maximum reflection loss of -44.08 dB at 9.56 GHz in X band with 3.10 mm thickness whereas -14.85 dB reflection loss at 17.75 GHz with 1.0 mm thickness in Ku band observed [20].

Very few groups of researchers using renewable precursor like Mukherjee, *et. al*. synthesized a radar absorbing CNF from plant fibre decorated with nickel nano particle having excellent MA property at 3mm thickness in the frequency range of 2-8 GHz. It showed 99% MA with -20 dB reflection loss having tap density of 0.04 $g.cm^{-3}$ and specific surface area of 640 $m^2gm.^{-1}$[21]. Mukherjee, *et. al*. synthesized a microwave absorbing CNM from plant fibre decorated with cobalt Nano particle and observed that as-obtained material exhibits excellent MA property at 4-5mm thickness in the frequency range of 2-8 GHz.

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It showed 96- 99% MA with -20 dB reflection loss [22].

The present work has been designed by using Taguchi optimization method, a statistical method, to minimize number of experiment as well as time. CNMs have been synthesized from plant fibres by pyrolysis method and decorated with metal nano particle to enhance of MA property with magnetic and reflection loss. as obtained CNMs were characterised and further study its MA application in Ku band of microwave spectrum.

Table 1: Taguchi optimization L-9 Orthogonal Array

Sr. No.	Sample	Cotton treatment.	Metal salt Treatment.	Temperature of pyrolysis.	Time Hrs.
01	L_1	NaOH	$Ni(NO_3)_2$	650^oc	2.0
02	L_2	NaOH	$Co(NO_3)_2$	700^oc	2.5
03	L_3	NaOH	$Cu(NO_3)_2$	750^oc	3.0
04	L_4	KOH	$Ni(NO_3)_2$	700^oc	3.0
05	L_5	KOH	$Co(NO_3)_2$	750^oc	2.0
06	L_6	KOH	$Cu(NO_3)_2$	650^oc	2.5
07	L_7	Untreated	$Ni(NO_3)_2$	750^oc	2.5
08	L_8	Untreated	$Co(NO_3)_2$	650^oc	3.0
09	L_9	Untreated	$Cu(NO_3)_2$	700^oc	2.0

II. EXPERIMENTAL

In this work, commercial grade of cotton fibres was used as precursor to synthesize CNM. All chemicals of AR grade were used for pre and post cotton fibres treatment and metal nano particles decoration on CNMs. The detailed procedure of CNM synthesis by pyrolysis method is discussed elsewhere [23-25].

Taguchi optimization L-9 Orthogonal Array having three levels and four factors. Accordingly, nine CNMs samples were prepared and labelled them as L_1, L_2, L_3 up to L_9, respectively. The first sample i.e. L-1 preparation and synthesis procedure according to table-1 is like cotton fibre was treated with NaOH solution and then washed with water. It was decorated with nickel nano particles and pyrolysed at 650^0C in presence of Argon gas for 2.0 hrs. Likewise other remaining 08 samples were prepared according to Taguchi optimization L-9 Orthogonal Array table. These samples were then used to study the reflection loss when incident with microwaves of 12 to 18 GHz, that is in the Ku band.

A. Characterization

The prepared sample were characterized by SEM, TEM, XRD, Raman Spectroscopy and MA (reflection loss). Characterization and morphological study were done with the help of scanning electron Microscope (SEM) Hitachi S-4300 instrument. Transmission Electron Microscopy (TEM) Philips, CM 200, operating voltage 20-200 kv, resolution upto 2.4 Ao, X-Ray Diffraction (XRD) study was carried out using - X'Pert Philips, range 2θ: 2.0000 <-> 80.0000°, radiation Cu-Kα, λ=1, 54056 Å. Raman Spectroscopy were carried out using Jobin Yvon Labram spectrometer and the laser excitation wavelength was 633 nm with a spectral resolution of <1.5 /cm. MA was measured with help of Vector Network Analyzer (VNA), using N5249A PNA-X Microwave Network analyser, 9 kHz -8.5 GHz.

III. RESULT AND DISCUSSION

A. Scanning Electron Microscope

The surface morphology and topography of synthesized carbon materials were characterized by SEM. The SEM images shows that the carbon samples have flakes like structure with crest on carbon material surface and the metal nano catalyst generated *in situ* are evenly distributed all over the surface of carbon having size ranging between 40-50 nm. CNM.

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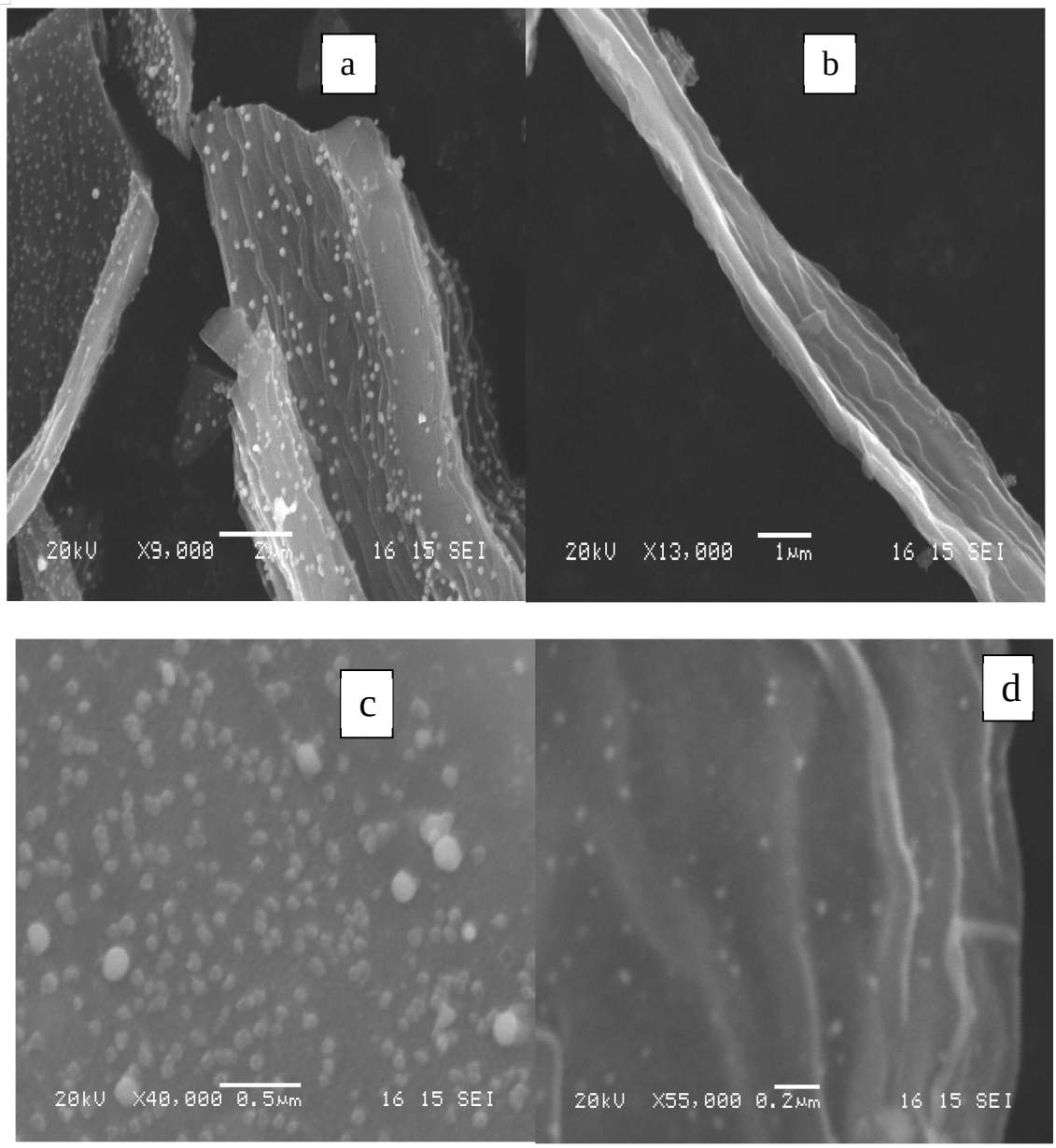


Figure: 1-(a) (b) and (c) SEM of L-1 sample and (d) SEM of L-6 sample

B. X-Ray Diffraction (XRD)

In figure 2, XRD graph of sample L-1 have been treated with nickel salt and XRD graphs of L-1 sample showing sharp peak at 2θ =11.58° is of Graphene oxide (GO), the broad peaks at 2θ = 23.64º indicates presence of Reduced Graphene oxide (RGO) [26-28]. Whereas, Sharp peak at 2θ = 44.56º, 2θ = 52.0^0 and 2θ = 76.5°indicates presence of nickel nano-particles while L-4, L-7 samples giving same observation because both the samples have decorated with nickel salt [29]. XRD Graph of sample L-2, sample L-5 and sample L-8, these samples have been treated with cobalt salt. L-8 graph shows sharp peak at 2θ = 10.3°, in L-2 peak at 2θ = 10.0°and in L-5 peak at 2θ = 11.7° is of Graphene oxide (GO) and the peak at 2θ = 23.5° indicates presence of Reduced Graphene oxide (RGO). Peak at 2θ = 44.5°and at 2θ = 47.3° indicates the presence of cobalt nano particle [30], L-3 , L-6 and L-9 samples these samples have been treated with copper salt and XRD Graph of these samples shows sharp peak at 2θ = 11.7° is of Graphene oxide (GO) and 2θ = 23.5° indicates presence of Reduced Graphene oxide (RGO). Peak at 2θ = 43.2°, 2θ = 50.3°and 2θ = 74.2° indicates presence of copper nano particles [31]. Whereas, peak at 2θ = 26.7° is of graphite persistent in all samples indicating presence of partly amorphous carbon [32].

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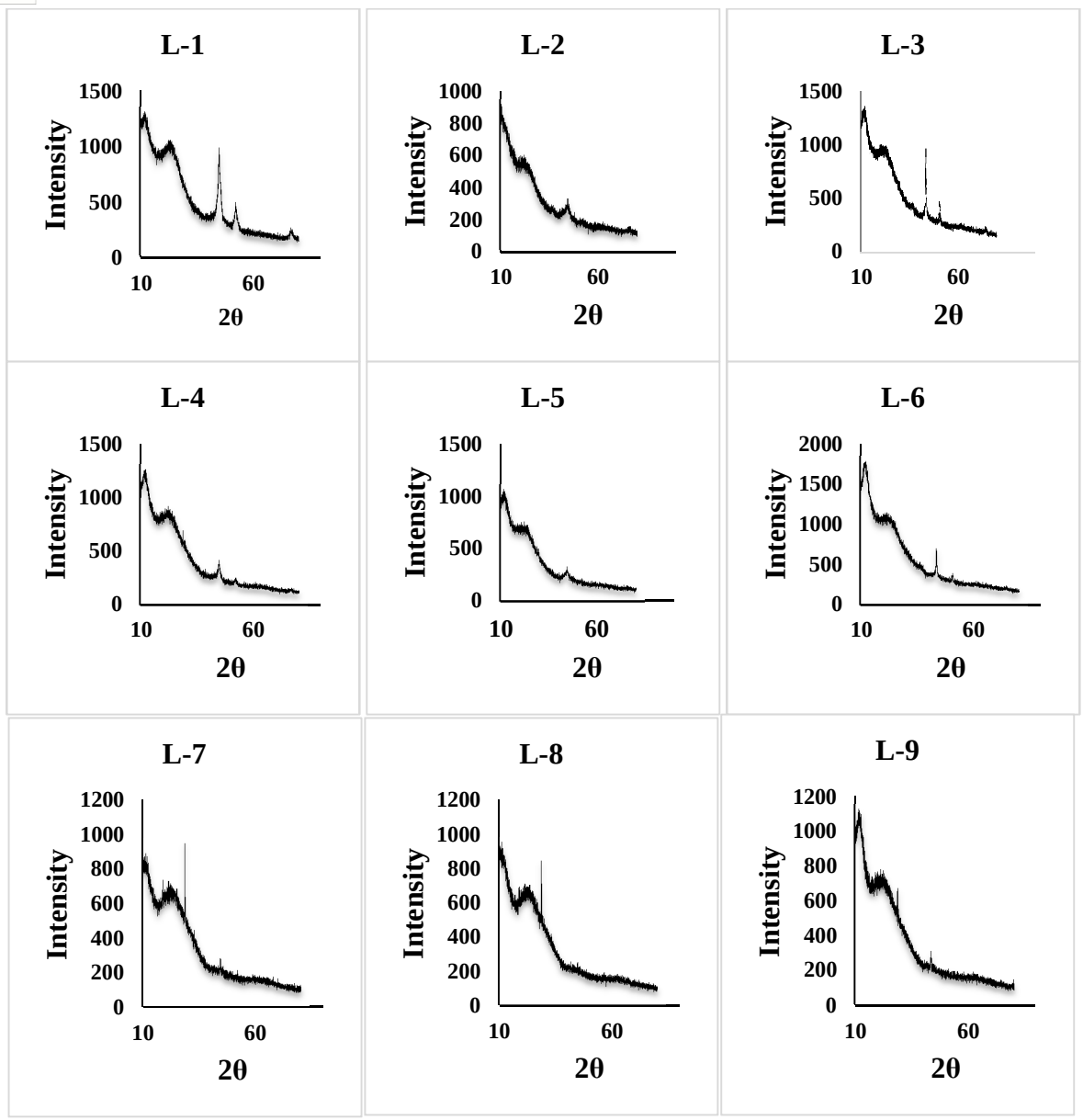


Figure: 2 - X-ray Diffraction of L-1, L-2, L-3, L-4, L-5, L-6, L-7, L-8 and L-9 sample.

C. Transmission Electron Microscope

Sample L-1 and L-6 were characterized by transmission electron microscopy, fig. 3 (a) image shows a carbon sheet edges like structures which is of 80-90 nm in thickness in length where as fig. 3 (b) sample shows the evenly scattered metal nano catalysts all over the surface having size of 40-70 nm are impregnated on the carbon surface.

D. Raman Spectroscopy

In figure 4, Raman spectrograph, L-1 sample having peaks at 1346 cm^{-1} D band and at 1589 cm^{-1} G band. The two peaks in the range of 1250 cm^{-1} -1650 cm^{-1} shows the presence of Reduced graphene oxide as well as disordered reduced graphene oxide [33] as the D band having higher intensity peak than G band. Sample L-3 has similar peaks as L-1 indicating presence of reduced graphene oxide.

L-8 sample shows similar peaks in the range of 1250 cm^{-1} -1650 cm^{-1} like L-1 and L-5 corresponding to D band and G Band. In this case, the G band has more intensity than D band therefore, the as obtained carbon sample is of graphene oxide. The broad peak in the range of 2700 cm^{-1} – 2900 cm^{-1} indicates the presence of amorphous carbon in the samples [34].

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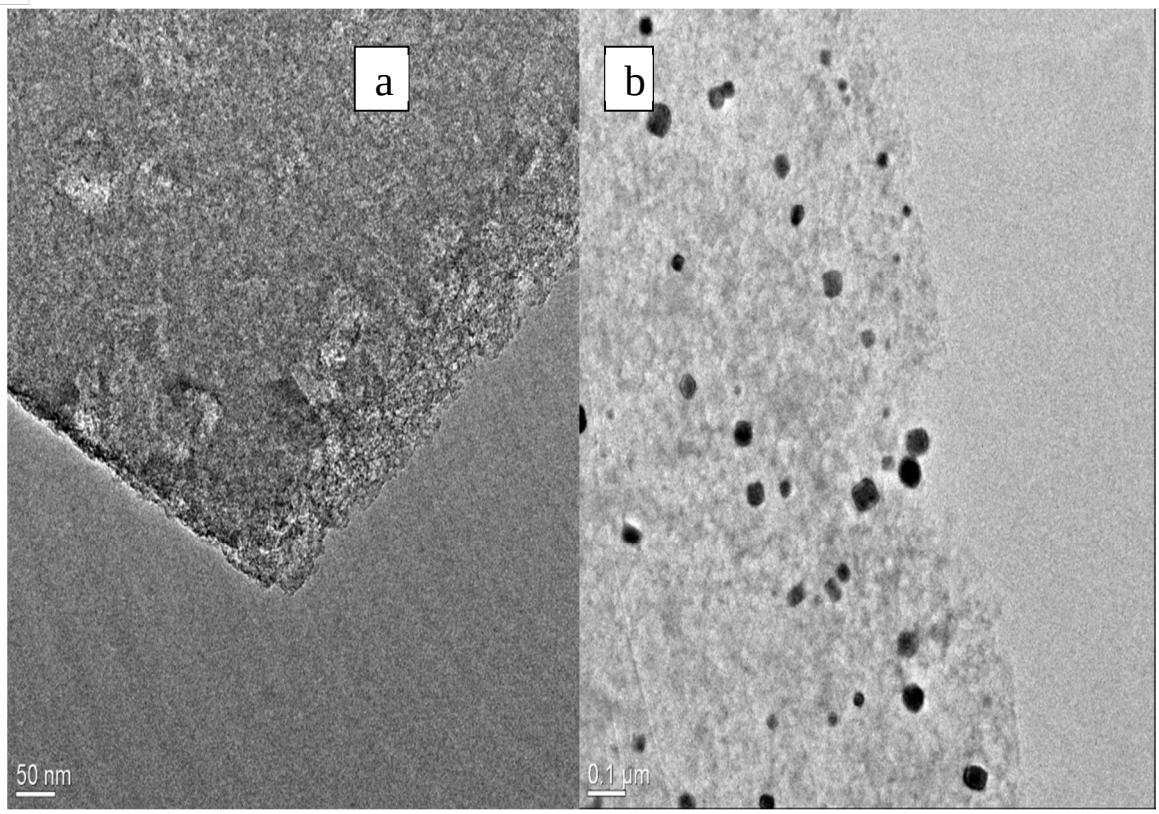


Figure: 3 – (a) and (b) transmission electron microscopy images of L-1 Sample and L-6 sample.

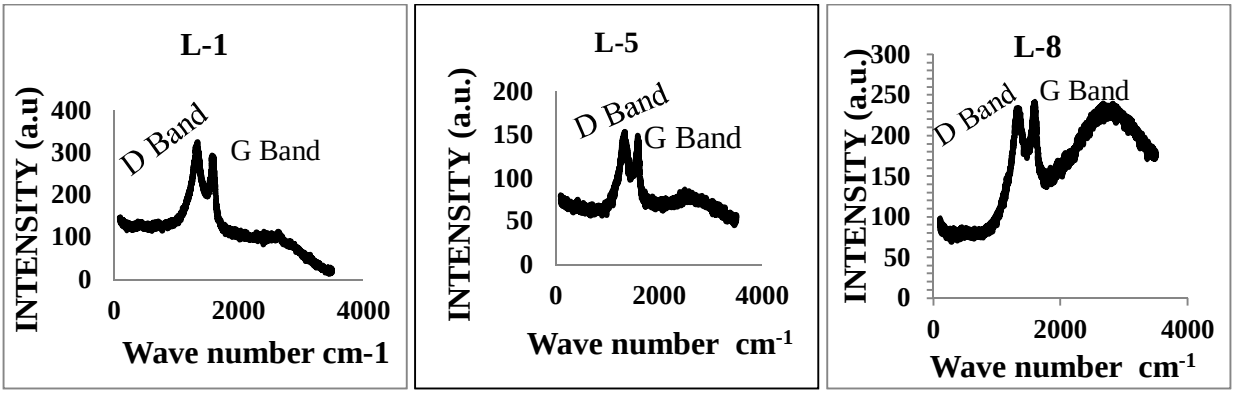


Figure: 4 – Raman spectrograph of L-1, L-5 and L-8 sample.

E. Reflection Loss (RL) Study

All these sample were analyzed by vector network analyzer for reflection loss study in 12 – 18 GHz i.e. Ku frequency band. Sample L-1 showing two major peaks, first peak at 14.61 GHz which is of -26.78 dB and the other peak observed at 17.67 GHz having -29.03 dB RL whereas -10 dB RL observed from 14 GHz – 15 GHz frequency range approximately 1 GHz bandwidth, same band gap observed between 17 GHz – 18 GHz frequency range with 1 GHz bandwidth. In sample L-2 high intensity peak observed, first at 14.31 GHz of -30.42 dB RL and another peak at 17.67 GHz of -33.51dB, whereas graph shows -10 dB RL from 14.07 GHz - 14.96 GHz and 17.01 GHz -18.0 GHz frequency range, here also 1 GHz bandwidth observed. Sample L-3 having same two peaks one at 14.37 GHz which shows -33.44 dB RL and another peak at 17.67 GHz with -34.78 dB, second peak having little higher intensity than first, while minimum -6 dB RL observed throughout the Ku band frequency range. The carbon sample L-4 have -32.30 dB RL at 14.34 GHz whereas -30.56 dB RL observed at 17.49 GHz with minimum -5.68 dB RL for whole 12- 18 GHz frequency range. Sample L-5 observed highest intensity peak of -37.11 dB RL at 14.31 GHz whereas -28.17 dB RL observed at 17.49 GHz with minimum -6.01 dB RL for Ku band.

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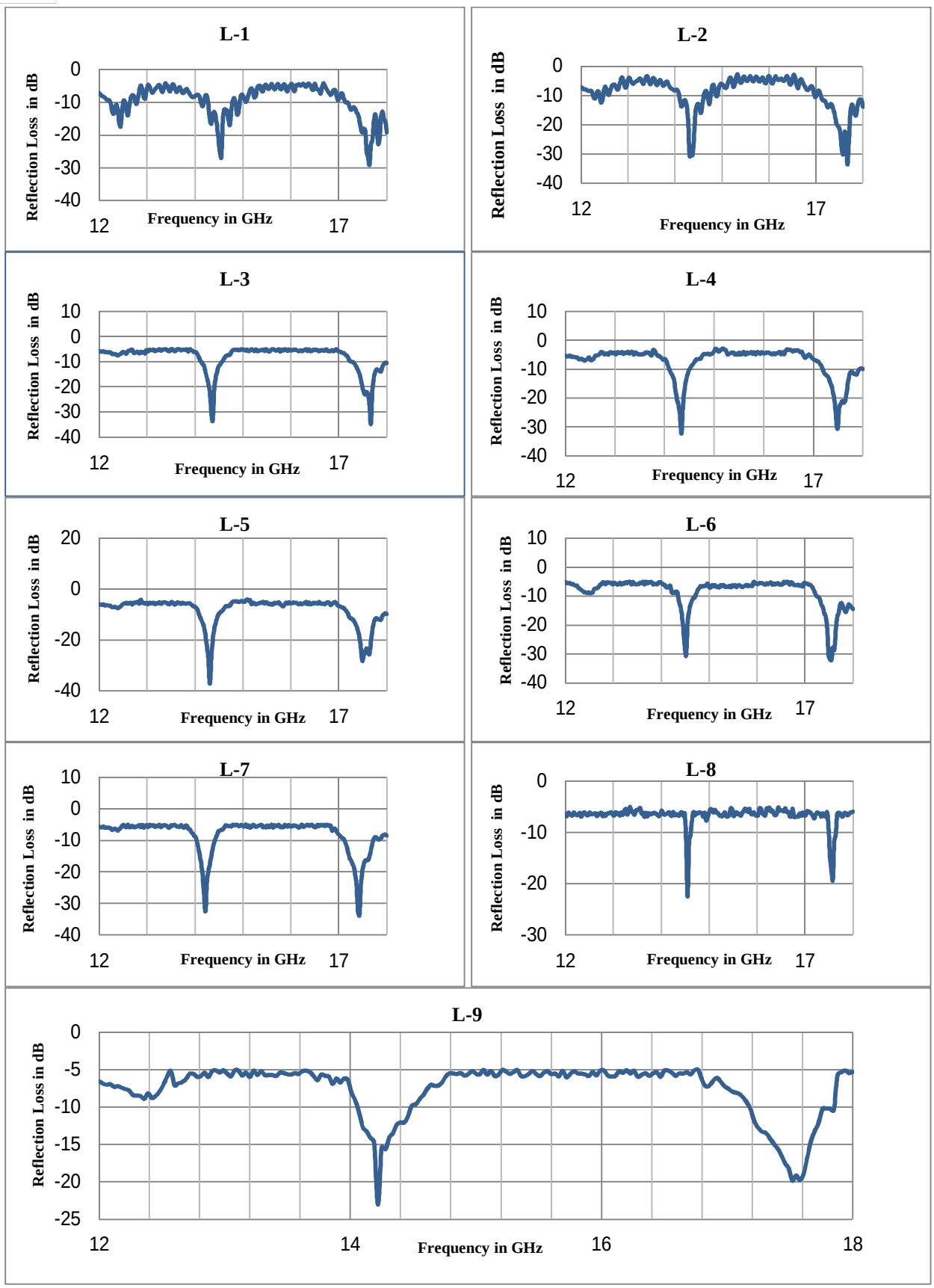


Figure 5: - Reflection loss graph of all nine-sample labelling as L-1, L-2, L-3, L-4, L-5, L-6, L-7, L-8 and L-9.

Sample L-6 observed -30.32 dB RL at 14.52 GHz and -31.85 dB RL at 17.52 GHz both peaks having nearly same intensity with minimum -5.64 dB RL for Ku band. The L-7 sample also having good RL intensity peaks like at 14.22 GHz observed -32.47 dB RL and at 17.33 GHz – 33.77 dB RL with -5.46 dB RL throughout the Ku band frequency range. in L-8 carbon sample sharp peaks observed at 14.55 GHz which is of -22.49 dB RL and -19.41 dB RL at 17.58 GHz both peaks are very sharp and having narrow band gap with less than 0.4 GHz bandwidth. Sample L-9 shows peaks at 14.37 GHz of -23.69 dB RL and -19.83 dB RL at 17.63 GHz, here first peak having higher intensity than another with -5.08 dB minimum reflection loss throughout Ku band frequency.

The mean of signal to noise ratio of the observations of reflection loss of the samples under study for 2.5 mm thickness of the composite indicates the best parameters that may be used to obtain a better outcome. The fig. 6 indicates the best parameters for synthesis of the carbon samples.

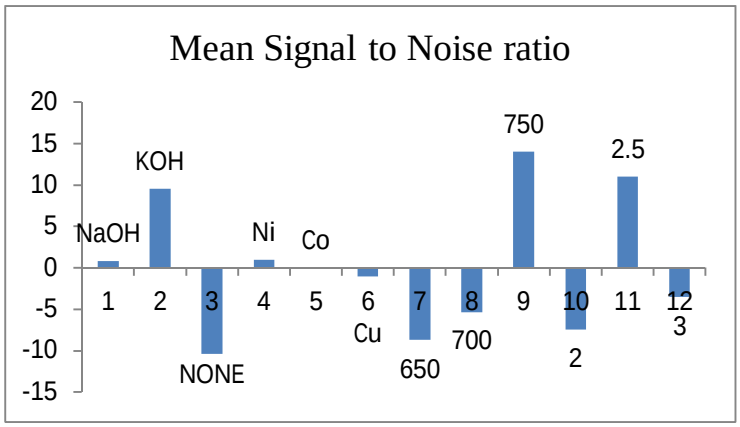


Figure 6. mean of signal to noise ratio

The best set of parameters as obtained from Taguchi optimization methodology is pre-treatment of KOH, metal ion treatment of nickel, temperature of 750ºC and time of heating to be 2.5 hours.

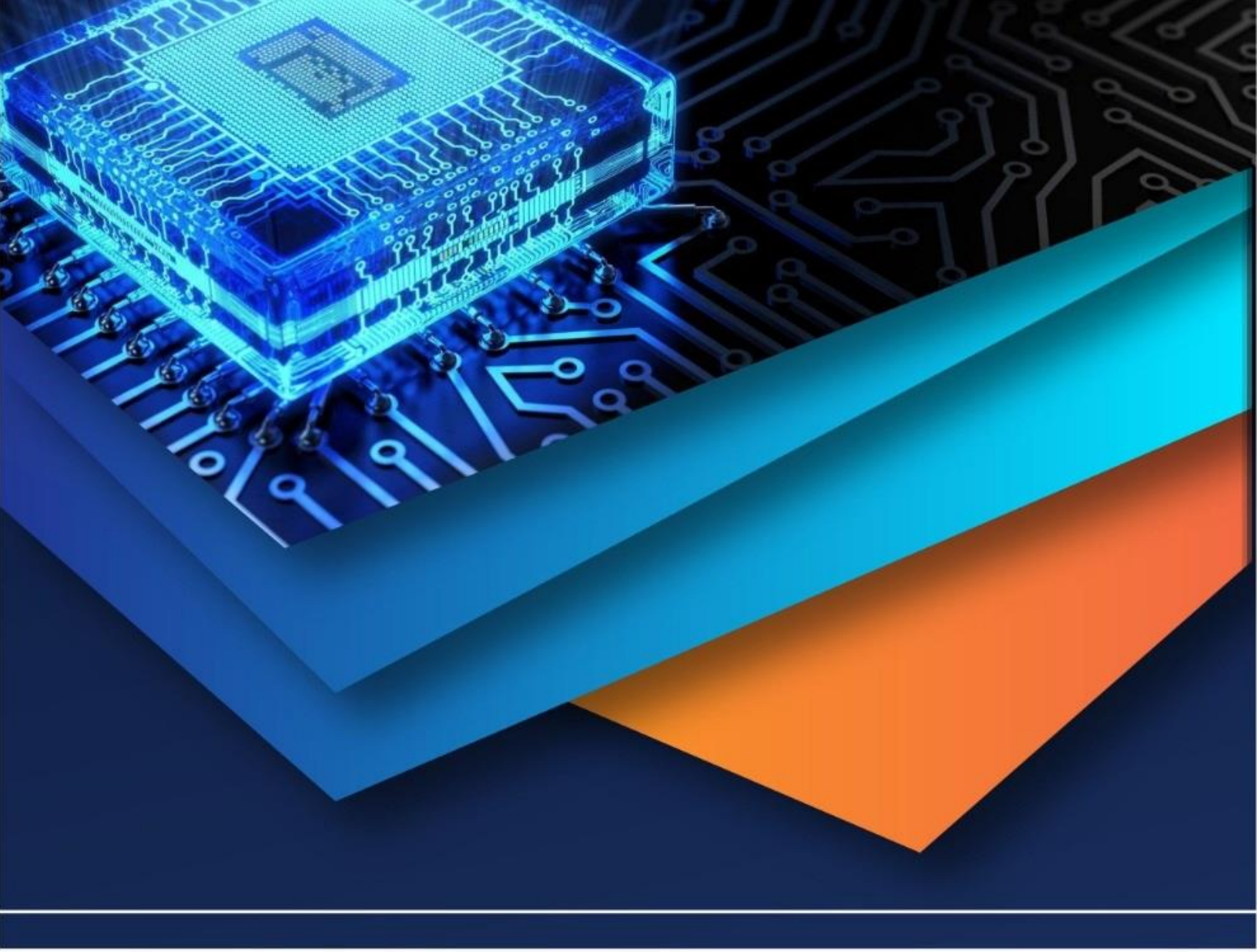
IV. CONCLUSION

Carbon materials synthesized from plant-based precursor by carbonization method using Taguchi optimization method to minimize repetition of experiments, synthesized Carbon materials having nano size confirmed by SEM and TEM. SEM images showed decorated metal nano particles having 40-50 nm in size whereas tem confirmed carbon materials having 80-90 nm in thickness. The prepared samples are mixture of amorphous and graphitic / crystalline in nature confirmed by XRD and Raman spectroscopy. All synthesized carbon nano materials having two distinct peaks at 14.5 GHz and 17.5 GHz, with an average RL more than -20 dB with 1 GHz bandwidth. Maximum RL observed -37.11 dB at 14.31 GHz and at 17.76 GHz RL observed -34.78 dB. whereas -5 dB RL observed throughout the Ku band frequency range. Therefore this could be a good microwave absorbing candidate for particularly 14-15 GHz and 17-18 GHz microwave frequency range bandwidth.

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