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Microwave Absorption Study of Carbon Nano Materials Synthesized from Natural Oils

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Abstract

Thin films of carbon-nano materials (CNMs) of different morphology have been successfully deposited on ceramic substrate by CVD at temperatures 800°C, 850°C and 900°C using plant based oils in the presence of transition metal catalysts (Ni, Co and Ni/Co alloys). Based on the return and insertion loss, microwave absorption properties of thin film of nanocarbon material are measured using passive micro-Strip line components. The result indicates that amongst CNMs synthesized from oil of natural precursors (mustered oil - *Brassica napus*, Karanja oil - *Pongamia glabra*, Cotton oil - *Gossipium hirsuta* and Neem oil - *Azadirachta indica*) carbon nano fibers obtained from neem's seed oil showed better microwave absorption (~20dB) in the range of 8.0 GHz to 17.90 GHz.

Keywords : *Microwave absorption, carbon nanofibers, Synthesis carbon fiber*

1. Introduction

Microwave absorbers have been widely used to prevent or minimize electromagnetic reflections from large structure such as aircrafts, ships and tanks. Magnetic materials such as ferrites, iron and Co-Ni alloys are used as absorbers [1, 2]. The main problem for the design of magnetic absorbers is related to the choice of the material. In addition, these materials require thick coating to meet practical demands. Recently scientists have started using carbon nanomaterials for studying microwave absorption properties [3-5]. Most of these works have been carried out with carbon materials derived from petroleum based precursors. Sharon *et al.* [6] has been developing plant based precursors for the synthesis of various forms of carbon nanomaterials and studied their applications. Recent observation has shown an appreciable microwave absorption by carbon nano-beads synthesized from camphor. But the absorption was studied in the range of 8 GHz to 12 GHz and they observed highest absorption at 9.5 GHz and 11.5 GHz. It would be interesting if we can develop carbon nano-materials which could absorb from 8 GHz to 18 GHz. The aim of this work was therefore to synthesis carbon nano materials from plant based precursors (mustered oil - *Brassica napus*, Karanja oil - *Pongamia glabra*, Cotton oil - *Gossipium hirsuta* and Neem oil - *Azadirachta indica*) and study their microwave absorption properties in the frequency range 8.00 GHz-18.00 GHz.

2. Experimental

2.1. Preparation of CNMs

A schematic diagram of the CVD unit with its details description is shown in Fig. 1. Precursor to be pyrolysed is kept in the boat (B) and catalyst powder (size ~40-50 nm) spread over the ceramic substrate is kept in boat (D). Catalyst particle of such size can be produced by the urea decomposition method as it is discussed in [6]. In order to remove air from the quartz tube (C), hydrogen gas (G) is allowed to pass through the quartz tube (C). The position of quartz tube (C) is arranged in the furnace (A) such that the boat (B) is almost near the end of the horizontal furnace.

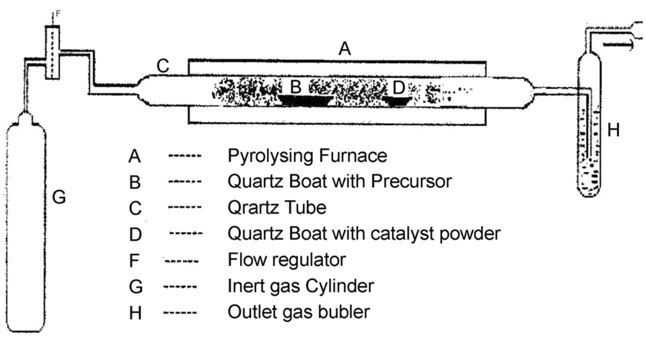
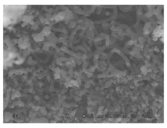
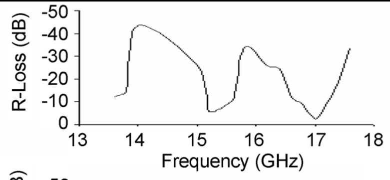
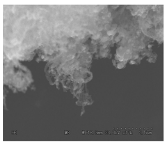
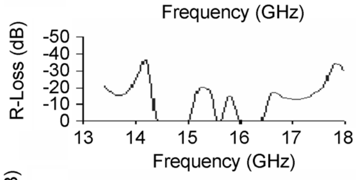
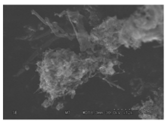
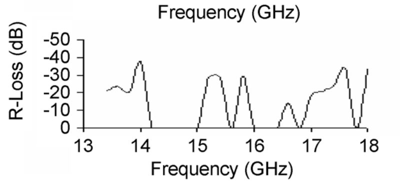
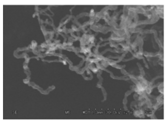
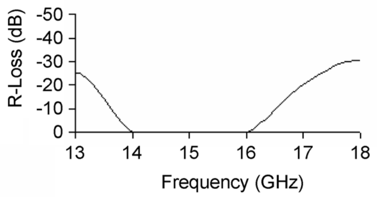
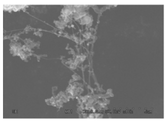
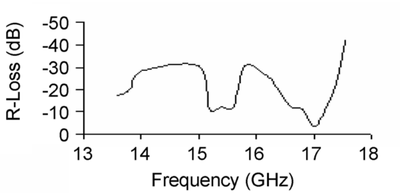
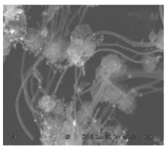
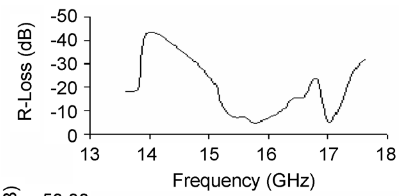
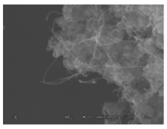
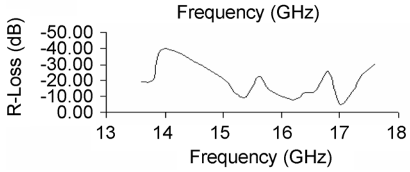
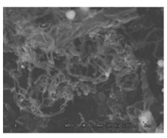
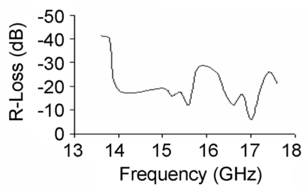


Fig. 1. A schematic diagram of the CVD unit.

This is done so that until the furnace reaches the desired temperature, precursor remains at temperature below its vaporizing temperature (i.e. ~100ºC). When the furnace attains the desired temperature, quartz tube (C) is moved towards the centre such that boat (B) experiences the temperature around 300ºC. At this temperature, precursor's vapour follows along with hydrogen gas towards the high temperature zone where it gets pyrolysed in presence of the catalyst kept on the substrate in boat (D). A thin film of carbon is deposited. When the entire vapour of precursor is pyrolysed,

Table 1. Details of the conditions under which different precursors were pyrolysed and their SEM micrographs are shown here.

No	Precursor	Temperature (ºC)	catalyst	SEM	Return Loss graph vs 13.0-18.0 GHz	Absorption Peak (GHz)
S1	mustard	850	Co			13.90, 15.80, 17.65
S8	mustard	800	Ni			13.90, 17.65
S7	mustard	900	Ni-Co			13.90, 15.30, 17.55
S6	karanja	850	Ni			17.60
S9	karanja	900	Co			13.90-14.90, 15.90, 17.40
S2	cotton	900	Ni			13.90, 17.90
S12	cotton	850	Ni-Co			13.90, 17.90
S10	neem	900	Ni			13.90-17.90

furnace is cooled down and the substrate is taken out for characterization. Atmosphere of hydrogen in the quartz tube is maintained through out the experiment. Thin films were analyzed by SEM. Details of the conditions under which different precursors were pyrolysed, and their SEM micrographs are shown in Table 1.

2.2. Measurement of microwave absorption

A microwave absorption by nano-carbon material was measured using micro strip line component with carbon nanomaterial as an overlay material. The proper impedance matching was done before the experiment was performed.To remove the effect of strip line the absorption with strip was noted before starting the microwave absorption study experiment. The Ku-band microwave bench was used to measure the microwave return loss before and after overlay. After impedance matching direct microwave input energy was measured. For the same input energy, return loss of microwave energy was measured for strip-line, substrate and different samples deposited on the substrate. Before plotting the graph of frequency versus return loss. The return loss by substrate was subtracted from the samples loss to remove the effect of substrate from sample.

3. Result and Discussion

A set of graph was plotted between frequency and return loss with and without sample on the substrate. Difference between return loss of substrate and the sample was then plotted versus frequency to get the amount of microwave absorbed by carbon nano materials at different frequencies. These graphs of the return loss are shown in Table 1. It is observed that carbon materials obtained from almost all precursors showed return loss at 13.90 GHz frequency (~ -30 dB). Some of them showed absorption at few other frequencies as well. In Table 1 the frequencies at which the absorption of microwave of magnitude greater than -30 dB occurred are given. These results suggests that there is little or almost no effect of temperature of pyrolysis and the type of catalysts used for the pyrolysis on the absorption of microwave frequencies in the range of 13.00-to 18.00 GHz, except with Karanja oil which gave higher absorption at 17.69 GHz for sample pyrolysed at 850^{o}C with Nickel catalyst. Carbon nano-materials obtained from mustered oil showed specific absorption at around 13.90 GHz, 15.80 GHz and 17.65 GHz (Fig. 2a). The carbon micro-fiber has diam meter of 2.5 μm (Fig. 2b).

However the carbon nano-fiber obtained with neem's seed oil gave absorption in the range of 10 dB to -25 dB for the frequency range 13.9 GHz to 17.9 GHz (Fig. 3a). Since carbon nanomaterials obtained from Neem's seed oil gave almost uniform absorption in the range of 13 GHz to 18

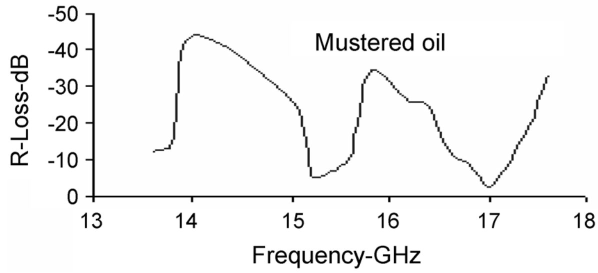


Fig. 2a. Return loss spectra obtained with carbon nanomaterial obtained by pyrolysis of mustered oil (Table 1).

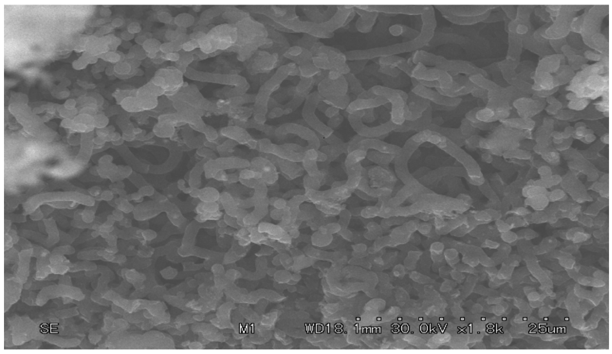


Fig. 2b. SEM micrograph carbon nanomaterial obtained by pyrolysis of mustered oil (Table 1).

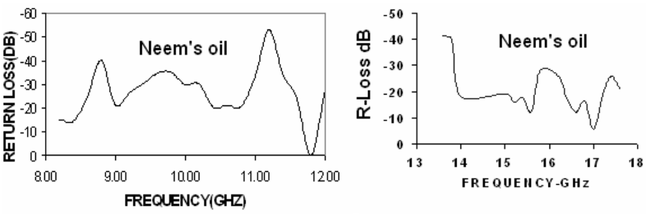


Fig. 3a. Return loss spectra of Carbon nanomaterials obtained from neem's seed oil (S-10, Table 1) (8-18 GHz) and (13 GHz-18 GHz).

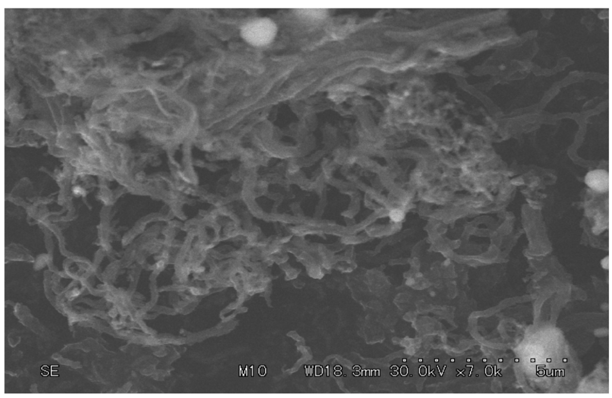


Fig. 3b. SEM micrograph of the carbon fibers obtained with pyrolysis of neem's seed oil.

GHz, return loss study of sample (S-10, Table 1) was also studied in the frequency range 8-12 GHz which showed return loss of almost -15 dB in the range of 8 GHz to 12 GHz and in specific a return loss of -21 dB in the frequency range 8.20 GHz-9.00 GHz (Fig. 3a). Absorption is found to suddenly improved upto -53 dB at 11.20 GHz and then drops to -23 dB at 11.60 GHz. This return loss study suggest that the carbon obtained from neem's oil can be a useful microwave absorber in frequency range 8.00 GHz-18 GHz with the return loss of around -15 dB. Efforts are now being made to improve its absorption further.

4. Conclusion

From graphs of different samples (Table 1) it is observed that sample having the morphology like nano fibers showed better absorption as compared to the other samples. It is suggested that carbon nanofibers obtained from neem's seed oil may be useful microwave absorber for the frequency range of 13.8 GHz to 17.90 GHz in ku-band and 8.00 GHz to 11.60 GHz in X-band.

From SEM image (Fig. 2b and Fig. 3b), it is observed the neem based fibres are having the rough surface, and soft in comparison with muster based fibers, which may help the absorption with maximum return loss. On the other hand mustered based fibers are smooth and hard which reduces the return loss due to maximum reflection of microwave energy. Also carbon obtained from mustered oil contains some amount of carbon nano-beads. In addition, the diameter of the fiber may be one of the parameter playing an important role in microwave absorption, as the neem based fiber with diameter of 250 nm shows better absorption than the mustered based fiber which has diameter 2.5 μm (Fig. 2b and 3b). From these results it could be concluded that for better microwave absorption, the carbon nanomaterials should have soft texture with small diameter.

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