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Microwave wide bandabsorption by carbon from Corn cob-1

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Abstract

Corn cob, an agricultural waste, is parolyzed at different temperatures (700°C, 800°C and 900°C). Microwave absorption of carbon in the frequency range of 2GHz to 8GHz is reported. Carbon activated with 5% nickel nitrate showed more than 90% absorption of microwave in the frequency range from 6GHz to 8GHz, while carbon activated with 10% Nickel nitrate treated corn cob showed 90% absorption in the frequency range of 2.5 GHz to 5 GHz. Carbon showing the best absorption are characterized by XRD, Raman spectra and SEM . It is suggested that corn cob treatment alone with 1N KOH did not improve the microwave absorption, whereas treatment along with nickel nitrate improved the absorption property much better. It is proposed that treatment with nickel nitrate helps in creating suitable pores in carbon which improved the absorption behavior because while treating carbon with 1N HCl helps to leach out nickel creating equivalent amount of pores in the carbon.

Key words

Corn cob Pyrolysis, microwave absorber, activated carbon, Broad band absorption

1.0 Introduction

Microwave absorbing materials have drawn lots of interest in commercial and military purposes fortheir applications in wireless communication, local area network, satellite, television etc., because they can be used to minimize reflectance of microwave from surfaces of aircrafts, ships, tanks, electronic equipment etc. Normally materials like ferrites, carbonyl iron have been used for absorbing microwave. But most of these materials are overweight and hence limits their utilization. It is contemplated that a composite of the magnetic materials with low density materials might improve the absorption properties. Therefore, lots of efforts are being made to develop materials that are relatively light in weight, corrosion resistant, structurally sound and flexible andwhich could absorb electromagnetic (EM) wave in a wide frequency band.

For application especially in aircraft, thinner absorbing material is required which could absorb more than 90% microwave (i.e. >-20dB) in the required frequency range. Carbon materials (carbon nanotube,carbon nano fibers, carbon nano-beads etc) are considered to suffice these requirements. But it is still an open question as to which form of carbon nanomaterials could be able to provide wide band width absorption of microwave. For improving the absorption properties, methods like mixing, doping and filling magnetic nanoparticles in CNT have been adopted.

Requirements for microwave absorbers in principle can be of twofold: absorber should be able to absorb specific frequency of the microwave to tune of more than 90% (i.e. > -20dB) or absorbers should be able to absorb a wide broad band width of frequencies to the tune of 90% which may be from 2 GHz -10 GHz, or 2 GHz – 18 GHz or 2 GHz -24GHz depending upon the requirement. Most of the publications are geared towards the former types of requirement; very little informations are available for the latter type of requirements. Zhong et al [1] studied microwaveabsorption byboron and nitrogen doped CNT composite with Fe_3O_4 in the frequency range 2GHz -18 GHz. Depending upon the thickness of material they showed the specific absorption in the range of 10GHz to 12 GHz with reflectionloss up to -40dB at 12GHz. Gu Lu et al [2] studied the whisker of carbon nanotube with ZnO in the frequency range of 2 GHz -18 GHz. They could show reflectance loss of -18dB for specific frequency around 12 GHz. It was interesting that by changing the composition of the whisker, the specific frequencies could be maneuvered to even 6 GHz and 9 GHz but with lower reflectance loss (~ -6 dB to -10 dB**).** On the similar line many others have studied the microwave absorption using composites of oxides with carbon material, which could absorb some specific frequency to the tune of even -40dB. They studied in the frequency range of 2GHz -18GHz. [3-7]

Composite with more than one metal (CuO/Ag/Carbon) has been used to study the microwave absorption in the range of 2 GHz– 18GHz and reported that depending upon the thickness of the material reflectance loss of -20dB could be obtained for frequencies 6 GHz,10 GHz, 12GHz and 14 GHz. It is interesting to note that reflection loss remains the same but the frequency range could shift depending upon the thickness of the sample [8].Xiaosi et al[9]have reported that even composite of nano size iron with CNT can be used to get microwave absorption in the range of -6dB to -40dB. However, the reflectance loss peaks are very sharp. Thus material can be used for absorbing specific frequency in the range of 2GHz to 18 GHz. Lei Liu et al [10]have shown that a hybrid material of carbon coil and carbon fiber can alone be used for the microwave absorption in the range of 2GHz- 28 GHz. They showed that there is no need to add any metal or metal oxide. It is reported that carbon fiber alone could show a reflection loss of-2dB at 4GHz. But a hybrid of carbon fiber and carbon coil improves the reflection loss to almost -18dB for frequency 11GHz.

Recently Lei Liu *et al* [11] have reported that helical carbon nanofibers coated with carbon fibers can show broad band reflection loss at -10dB covering 8GHz to 18 GHz. In addition this material can also show reflection loss of -32dB at 9GHz.

Zhao *et al* [12] observed that PAN based carbon fibers can also show a broad band reflection loss of -10dB at frequency 9GHz- 15 GHz and -30dB at specific frequency of 11.5 GHz. Carbon derived from PAN/PMMA could also show broad band reflection loss of -5dB from 8 GHzto13 GHz frequency [13]

From these literatures it appears that broad band reflection loss covering wide frequency are reported at -5dB and -10dB, though some specific absorption were obtained of higher values (to the tune of -30dB also). This is also obvious from these observations that carbon material can be the best choice for developing microwave absorbing material.

Therefore, there is aneed to develop carbon material which could show broad band width reflection loss to the tune of more than – 20dB to -30 dB (i.e. > 90% absorption). In this report we are presenting our efforts on microwave absorption in the range of 2 GHz to 8 GHz by using carbon fibres synthesized from agricultural waste corn cobs. In order to improve the microwave absorption effect of chemical activation of carbon fibres are also presented.

1.1 Application of agricultural waste material for microwave absorption

Most of the work carried out with carbon material issynthesized from precursors derived from the fossil fuel. This makes the final material expensive. In the event of the fossil material being depleted, a vacuum could be created for want of precursors. Itis therefore, necessary to develop carbon fibers synthesize from precursors which are agricultural based. Because agricultural and forestry wastes, like coconut fiber, corn, baggas etc, are inexpensive and renewable. These waste materials can become a problem for their disposal. Considering these advantages of agricultural waste, Azizah Shaaban *et al* [14] synthesized carbon from rubber wood dust and prepared a composite with polyurethane matrix. They used this material to study the microwave absorption of frequency range 1GHz to 3GHz. They reported about 7 absorption peaks ofreflectanceloss of -8dB with the exception of one peak at 1.5 GHz giving -16dB.Sharon and his research group [15-17] are the first one to have developed techniques to synthesize all types of carbon materials from such agricultural waste.Dattatray *et al* [18-19] studied microwave absorption in the frequency range from 12GHz to 18GHz showing reflection loss less than -25dB (i.e >98% absorption) by using carbon nanomaterials synthesized from mustered oil and Karanja oil. Corn cob being an agricultural waste, the present report gives an account of the efforts made to synthesize carbon fiber from corn cob and chemically activate to study its application as microwave absorption in frequency range of 2GHz to 8 GHz.

1.2 Corn cobs for microwave absorption

Maize known as *Zea mays* is a grain crop. The leafy stalk produces ears which contain seeds called kernels. Maize kernels are used as vegetable, flour, grain and also for starch. Ears from where the kernels are removed are known as corn cobs which are considered as a waste product of maize. United States Department of Agriculture (**USA**) estimates that the World Corn production in 2013/14 was around 967.52 million metric tons, whileIndia produced around 230million metric tons in the same year.

Presently most of corncobs are either burnt or thrown in the river or used in place of coal with $1/3^{rd}$ of coal value [20]. Corn cob parolyzed[21-24]after treatment with 1N sKOH produces activated carbonmaterial which possesses surface area between 250 and 2410 m^2/g and pore volumes between 0.022 and 91.4 cm^3/g. The variations in the values are due to type of chemicaltreatment and the variation inparalyzingconditions. It is interesting to note that during the pyrolysis at 600^oC three types of products are formed; liquid, gas and solid [25]. These products have been analyzed. It is reported that the liquid products (~34- 40.96% wt),contains, phenols, 2-furanmethanol, 2-cyclopentanedione, the gas products (27-40.96% wt) consists of CO_2, CO, H_2, CH_4, C_2H_4, C_3H_6, C_3H_8 and the solid carbon material (23.6-31.6% wt). Based on these results and considering the quantity of maize being produced annually in the world, activated carbon can be produced to the tune of approximately 200 million metric ton annually. It is therefore important that scientists develop some useful applications of carbon produced from corn cob so that this type of research not only solve the problem of disposal of corn cob but also upgrade the waste product into a value added product. It is important to realize that disposal of corn cob should be completed within 6 months, otherwise it gets infected.

Carbon from corn cob has been used for adsorbing methane gas. Methane storage capacity [26] was found to be 160 (v/v) at 298 K and 1500 psi . Corn cob can be digested to generate electricity [27-30].

We have developed a method to utilize corn cob to study its capacity to absorb microwave in the frequency range of 2GHz to 8 GHz. To the best of our knowledge there has been no report published on the microwave absorption by carbon derived from corn cob in the frequency range 2 GHz to 8 GHz. These frequencies are selected because they are used for communication purposes. Under this paper the application of carbon obtained by the pyrolysis of corn cob as microwave absorber is discussed.

2.0Experimental

2.1Materials and methods

Dry stalks of maize (*Zea Mays*) were collected (Figure1),washed with slightly warm water to remove any mud or dirt and dried at 60^{o}C for 2h.

Figure 1 (A) maize crop (B) Leafy stalk produces ears which contain seeds called kernels (C) Maize kernels removed from (B) is referred as corn cobs,(D) Dried corn cobs powder after treatment with 1NKOH and (E) Dried powder of (D) after soaking with 10% $Ni(NO_3)_2$ 6(H_2O) solution.

Dried corn cobs were soaked overnight in 1N KOH solution at room temperature in the ratio of 1:2v/v.After 1day these corn cobs were filtered and washed till neutral pH. This treatment was done to remove some of the lignin and improves the surface area of carbon [21-24]. The residue (Figure 1D) was dried in a muffle furnace at 100^{o}C for 4h. In order to improve the absorbing capacity of carbon, Corn cob treated with 1N KOH was further soaked with nickel nitrate solution. For this purpose, the dried material was divided into three equal parts.To each portion (10g) aqueous solution (20ml) of nickel nitrate hexahydrate salt($NiNO_3.6H_2O$) of concentration 1% ,5% and 10% w/v (Figure 1E) were added respectively. The amount of nitrate solution was adjusted such that it was sufficient to soak the entire corn cobs. These mixtures were left overnight for soaking. Latereach fraction was heated in a muffle furnace for 2 hours at 100^{o}C to get dry powder. These powders were used for the pyrolysis. The purpose of nickel nitrate treatment was to create some pores into the carbon fiber produced from corn cobs fiber. It was expected that site at which nickel is adsorbed in corn cobs,after the pyrolysis the site will contain NiO. By treating pyrolysed corn cobs with 1N HCL all NiO will dissolve leaving behind pores equivalent to its size.

2.2 Pyrolysis of corn cobs

Unit of pyrolysis instrument is shown in Figure 2. This unit comprises of a horizontal furnace (A) and a ceramic tube (B). Quartz tube (C)is inserted into the ceramic tube (B). Quartztube (C) also contains a quartz boat (D) onto which treated corn cobare kept for the pyrolysis. Quartz tube (C) is connected with a gas cylinder (H). Gas flow is controlled by valve E and F.Another gas flow (G) is connected to the quartz tube (C) for allowing excess gas to escape from the system. Known weight of treated corn cobs is kept in the quartz boat (D).Quartz tube is flushed with nitrogen gas to make atmosphere free of oxygen. The furnace is switched on after setting the required temperature. Pyrolysis is carried for 2h (under flow of nitrogen gas) after the temperature has attained the required temperature. After completion of 2h furnace is switched off and carbon material is removed from the quartz tube when furnace has reached the room temperature. Pyrolysis of treatedcorn cob are carried out for three temperatures: 700^{o}C, 800^{o}C and 900^{o}C as per the condition mentioned in Table-1.

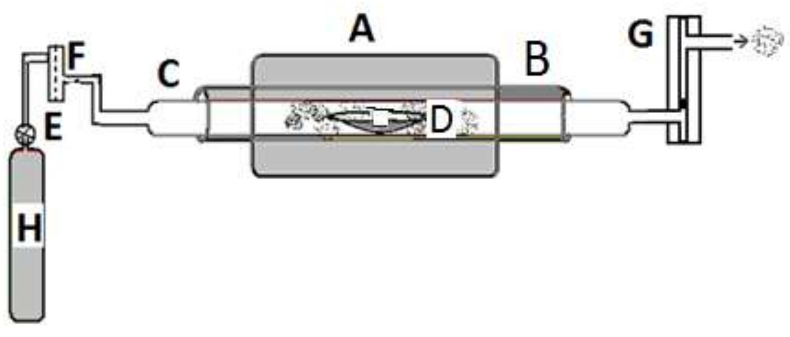

Figure 2 Schematic of a pyrolysis unit: (A) furnace, (B) ceramic tube, (C) quartz tube, (D) quartz boat, (E) gas regulator valve (F) flow meter, (G) flow meter for out let gas and (H) gas cylinder

Table-1 Experimental set up giving the details of the different condition of pyrolysis and results of microwave absorption in frequency range 2 GHz to 8 GHz.

Experiment No	Temperature (°C)of pyrolysis	1N KOH treated corn cobs soaked with Nickel nitrate solution (percentage)	Frequencies (GHz) at which absorption recorded was >70%
L1	700	1	4
L2	700	5	6 - 8
L3	700	10	2.5 - 4
L4	800	1	5
L5	800	5	6 – 8
L6	800	10	3 - 6
L7	900	1	3 and 7
L8	900	5	6 - 8
L9	900	10	4 - 6

The material obtained after pyrolysis wascrushed to very fine smooth powder. Crushed powder was kept in 1N HCl aqueous solution and sonicated for 30min and kept overnight. Finally the solution was filtered. The residue was washed repeatedly till neutralpH.This process dissolved all NiO which was adsorbed on corn cobs fibers (i.e. carbon fiber produced after pyrolysis). The solid residue obtained after filtration was dried at 80°C. This solid residue hence forth referred as "corn cob carbon-A (corn cob treated with 1%Ni), corn cob carbon-B (corn cob treated with 5%Ni) and corncob carbon-C (corn cob treated with 10% Ni).Pellets were made with fixed weight of corn cob carbon A, B and C powder. Pellet was used to measure the microwave absorption for the frequency range 2GHz to 8GHz

2.2 Microwave study

Wave guide technique was used to measure the absorption of microwave by carbon material. For these measurements a microwave test bench was setup that could cover the entire 2GHz to 8GHz (Figure 3). Return loss was measured in standard microwave waveguides. Power was measured by a Nd thermistor. Microwave source was the Gunn diode in the waveguides. Readings for the attenuated signal were taken by power meter. Return loss was calculated as % absorption.

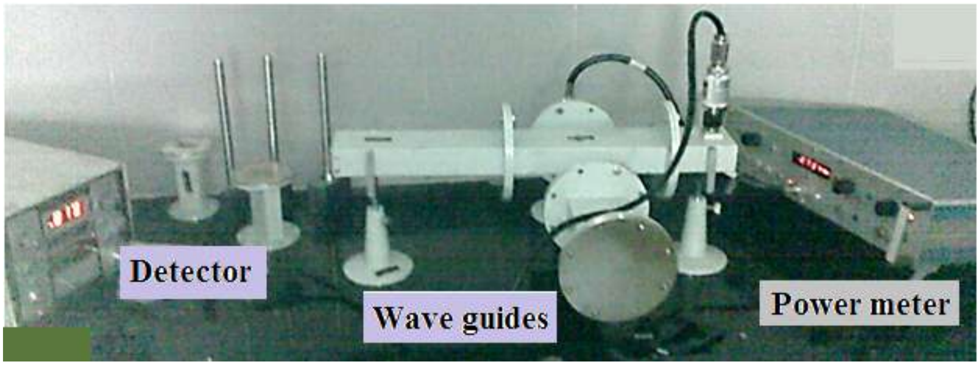

Figure 3 Microwave set up showing the arrangement of wave guide with power meter and the detector for the reflected wave

In the wave guide, first copper plate was kept and its return loss was measured. After this, over the copper plate, pellet of carbon material (corn cob carbon A, B and C) was kept and its return loss was measured. Difference betweenthe two returns losses was calculated to get the percentage of microwave absorption. This measurement was carried out with different frequencies (2GHz to 8GHz). Finally a graph was plotted between the percentage absorption and the correspondingmicrowave frequencies. Results are shown in Figure 4A-C.

3.0. Results & Discussion

Absorption percentage obtained with all nine experiments (Table-1) are shown in Figure 4A, B& C,where Figure 4A shows the result obtained with corn cobs carbon A, Figure 4B shows for corn con carbon –B andFigure 4C shows the result of corn cob carbon -C.Examination of these results suggests that there is a small effect of temperature of pyrolysis on absorption of microwave in the range 2GHz to 8GHz. However, there is a distinct effect observed with carbon obtained after soaking corn cob with different amount of nickel added to corn before pyrolysis and leaching out NiO from carbon after pyrolysis with 1N HCl..

Since the main object of this work was to synthesize carbon material which could absorb microwave frequency of more than 90% and absorption should cover wide band over a larger frequency range, the results of microwave measurements are examined from this angle.

3.1 Corn Cob-carbon –A (Carbon produced from corn cob soaked with 1% Ni before pyrolysis and carbon treated with 1NHCl after pyrolysis)

Results of microwave absorption obtained with corn cob carbon-A (Figure 4A) shows that carbon produced under conditions L1, L4 and L7 (table-1) do not show any wide band absorption in the range of 2GHz to 8 GHz. However, a specific 50% absorption peak for 4GHz is observed with carbon produced under L1.There are some absorption peaks of lower percentage. Similarly carbon obtained with condition L4 gives 80% absorption for the specific peak at 5GHz and others are at lower percentage. Carbon produced under condition L7 also shows 70% absorption for 3GHz and 7GHz frequencies. It appears that corn cob soaked with1% Ni and after pyrolysis carbon treated with 1N HCl isnot effective to show absorption of wide band frequency in this range i.e. 2GHz to 8GHz.

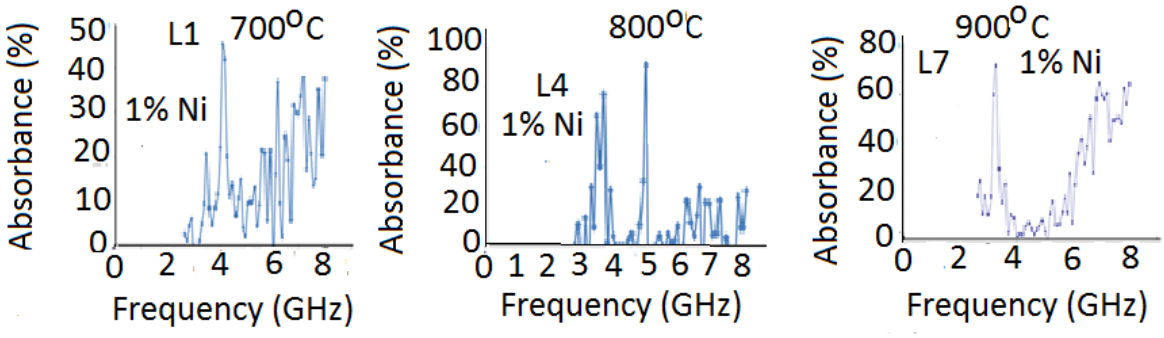

Figure 4A Graphs of %absorption versus microwave frequency obtained with carbon material obtained by the pyrolysis under the condition given in Table-1(L1,L4 and L7)

3.2 Corn cobs Carbon-B (Carbon produced from corn cob soaked with 5% Ni before pyrolysis and carbon treated with 1NHCl after pyrolysis)

It is interesting to observe that corn cobs carbon-B (Figure 4B) pyrolysed at 700°C, 800°C and 900°C (L2, L5 and L8) show a wide band absorption of more than 80% in the frequency range 6GHz to 8 GHz. Moreover, with increase in the temperature of pyrolysis the magnitude of percentage absorption of microwave frequency also increases. Carbon obtained by pyrolysis at 700°C and 800°C shows 80% absorption of 6 GHz to 8 GHz frequencies while carbon obtained by pyrolysis at 900°C shows 100% absorption of 6GHz to 8GHz frequencies(Figure 4B). However, broader width of absorption is obtained with carbon pyrolyzed at 800°C**.**

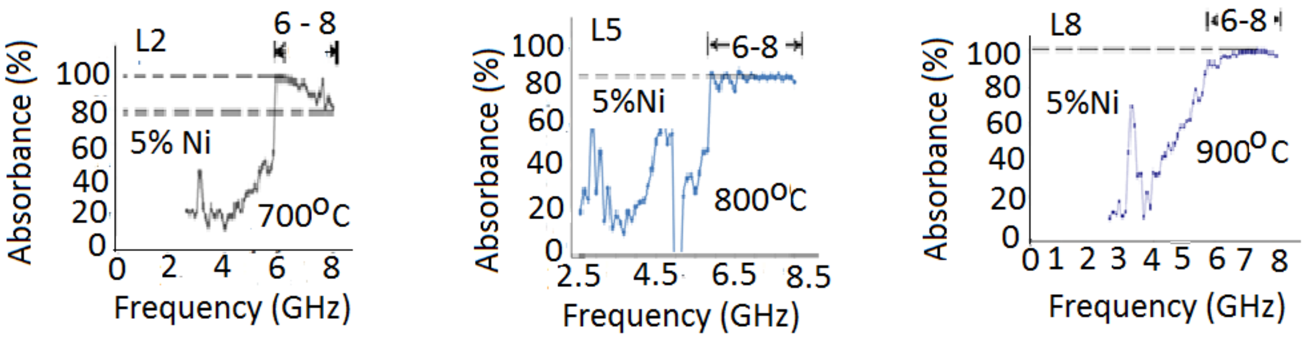

Figure 4B Graphs of % absorption versus microwave frequency obtained with carbon material by the pyrolysisunder the condition given in table-1(L2, L5, L8)

3.3 Corn cobs Carbon-C (Carbon produced from corn cob soaked with 10% Ni before pyrolysis and carbon treated with 1NHCl after pyrolysisl)

Carbon produced by pyrolysis of corn cobs carbon-C also gives wide band microwave absorption but at several frequencies.Carbon produced under L3 (Figure 4C) shows 100% absorption for 2.5 GHz to 4 GHz, while carbon with L6 condition shows ~ 70% absorption in the frequency range 2.5 GHz to 5 GHz. Carbon with L9 condition shows ~ 80% absorption in the frequency range 4GHz to 6 GHz.

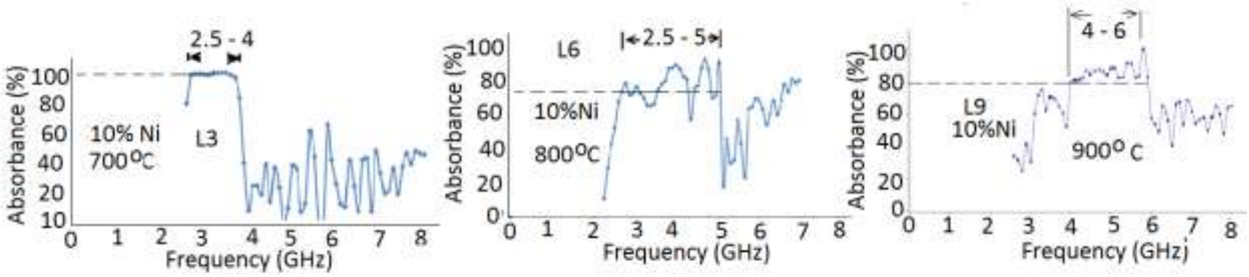

Figure 4C Graphs of % absorption versus microwave frequency obtained with carbon material by the pyrolysis under the condition given in table-1(L3, L6 and L9)

3.4 Effect soaking corn cob with Ni-nitrate and after pyrolysis treatment of carbon with 1NHCl

Soaking nickel nitrate with corn cobs and then pyrolysing at temperatures (700^{o}C, 800^{o}C and 900^{o}C) under nitrogen gas would produce NiO attached to the carbon fibers. It is expected that when this fiber is treated with 1N HCl for overnight, all NiO would get converted to $NiCl_2$ and since this salt is soluble in water would leach out from the carbon fibers. This conclusion is supported by the fact that, XRD of carbon prepared even with corn cobs soaked with 10% Ni-nitrate showsabsenceof Ni or NiO (Figure 5a). This suggests that if corn cob treated with 10% Nickel nitrate and after pyrolysis carbon treated with 1N HCl shows absence of Ni or NiO then corn cobs treated with lower concentration of nickel nitrate would certainly not contain any Ni or NiO. In addition, none of the SEM micrographs of carbon show any presence of nickel particle in the carbon (Figure 6).In other words, treatment with 1N HCl overnight has completely leached out all Ni or NiO from carbon.Therefore, it canbe concluded that leaching nickel from carbon may be creating equivalent amount of pores in carbon. These pores would thus be responsible to show better microwave absorption. Pores created with 1% Nickel nitrate may be not so large to show any useful effect on the microwave absorption. A similar observation has also been reported by Gu Liu *et al*[2], Lei Liu [10]Guang*et al* [13]and Ifeanyi *et al*,[31].

3.5 XRD and Raman spectra of carbon

Since carbon produced by L3 (Table-1) condition showed 100% microwave absorption for 2.5 GHz to 4 GHz range, its XRD and Raman spectra were taken (Figure5).XRD spectra shows (Figure 5a) two broad peaks corresponding to graphitic carbon of (002) and (100) planes. Moreover it does not show any other peak, suggest that carbon after treatment with 1N HCl has removed all nickel from the carbonmatrix and is graphitic in nature. Raman spectra (Figure 5b) also suggest that this carbon contains graphitic defect peaks at 1345 cm^{-1} (corresponding to D-band) and graphitic (G-band) at 1588 cm^{-1}.Guang et al [13] also observed that carbon fiber loaded with porous carbon shows higher magnitude of absorption, though it contained no metals like iron.

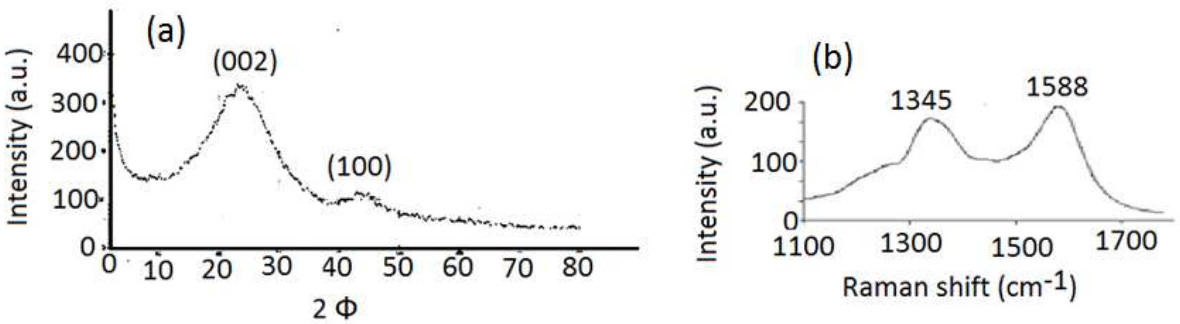

Figure 5 (a) XRD spectra of carbon produced under the condition L3 (Table-1) and (b) Raman spectra of carbon produced under the condition L3.

3.5 Morphology of carbon material by SEM studies

SEM micrographs of carbon materials obtained from corn cob synthesized as per conditions shown in Table-1 were taken and are shown in Figure 6A,B and C. Examination of SEM micrographs obtained with 1%Ni (Figure 6A)show that the morphology of carbon is like smooth plate and has almost nil fiberous structure. These carbon materials absorb microwave of a specific frequency in the range of 3GHz to 5 GHz.

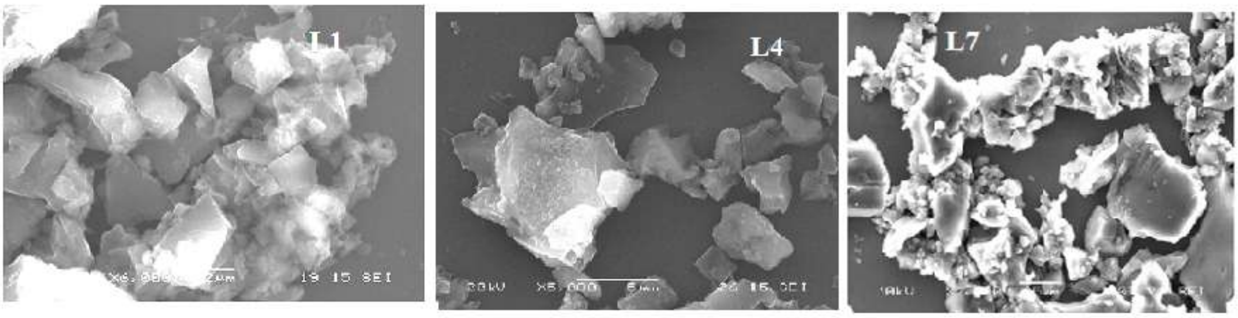

Figure 6A SEM micrographs of carbon obtained from corn cob prepared as per the Teble-1(L1, L4 and L7).Corn cob was treated with 1%Ni

Micrographs of carbon obtained with 5% Ni show fibrous type structure (Figure 6B) and this carbon also exhibit microwave broad band absorption greater than 90% in the range of 6GHz to 8GHz.

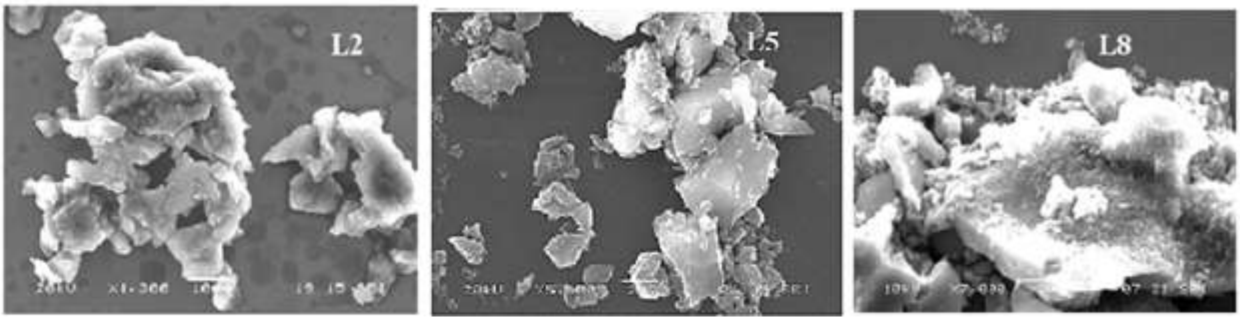

Figure 6B SEM micrographs of carbon obtained from corn cob treated with 5% Ni and pyrolysed as per the condition mentioned in Teble-1(L2,L5 and L8).

On the contrary, carbon obtained with 10%Ni though shows broad band absorption but only in the lower frequency range 2.5 GHz to 5 GHz (Figure 6C).The morphology of these carbons are also like large plate with very small fibrous nature. From these examinations it can be concluded that for observing broad band absorption in higher frequency range carbon should possess fibrous structure while for lower frequency range (3 – 5GHz) large plate type structure is suitable

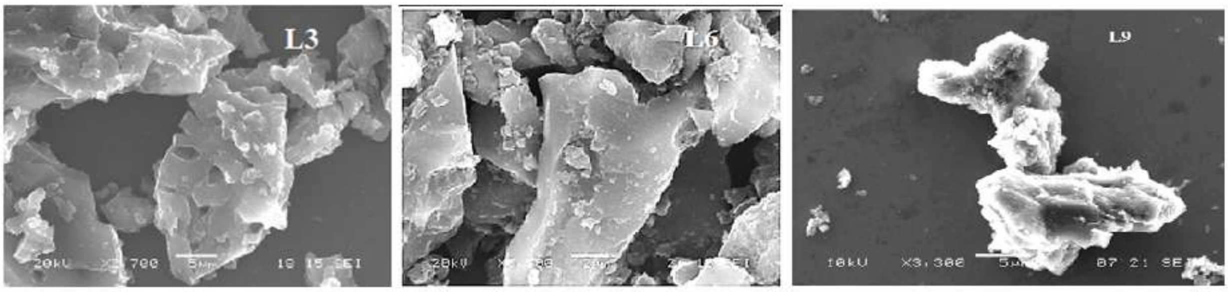

Figure 6CSEM micrographs of carbon obtained from corn cob treated with 10% Ni and prolysed as per the condition mentioned in Teble-1(L3,L6 and L9).

4.0 Conclusion

Carbon from corn cobs were obtained after treating it with 1NKOH and then activated with nickel nitrate of different concentration (1, 5 and 10 w/v%). Nickel treated corn cobs were pyrolyzed at 700^{o}C , 800^{o}C and 900^{o}C. Microwave absorption studies were made using wave guide techniques. It is observed that none gave uniform absorption in the range of 2GHz to 8 GHz. However, it is observed that almost 90% absorption is obtained for frequency range from 6GHz to 8GHz with carbon corn cob treated with 1NKOH and 5% nickel while for 2.5GHz to 5GHz frequency with 10% nickel treatment giving almost 90% absorption. It is also concluded that pyrolysis at 900^{o}C gives better results as compared to lower temperature. It is concluded that nickel do not directly affect the microwave absorption, but it is due creation of pores into carbon matrix.

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