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Optimization of parameters by Taguchi optimization method and synthesis of carbon nano materials (CNM) from oil rich non-edible seeds using chemical vapour deposition technique

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ABSTRACT- Present work describes the synthesis of Carbon Nano Materials (CNM) from oil rich non-edible seeds using CVD method and their purification by standard acid treatment. Optimization of parameters for maximum yield of CNM was done with the help of Taguchi method. There were three different precursors i.e. oil rich non-edible seeds (Ricinus communis, Madhuca indica and Azadirachta indica), temperatures for pyrolysis (800^oC, 900^oC and 1000^oC), carrier gases (Argon, Nitrogen and Hydrogen) and duration of pyrolysis (1, 2 and 3hrs). As per results obtained and Taguchi calculations the most effective parameter that impacted the CNM yield was the precursor (80.30%) followed by temperature (10.85%), then the duration of pyrolysis (8.50%) and the least effective parameter was carrier gas (0.35%). Impact of parameter on the yield is described by the deviation of signal to noise (S/N) ratio. The result of deviation of S/N ratio shows Azadirachta indica seeds were the best precursor, 800^0C was the most suitable temperature for duration of 1 hr and Ar was the best carrier gas for synthesis of CNM from seeds. The SEM micrographic study suggested that the seed derived CNM from Ricinus communis showed smooth plate structures with small fragments, while the seeds of Azadirachta indica and Madhuca longifolia derived SEM micrograph of CNM showed rough and rippled plate surfaces. The CNM from Azadirachta indica showed multilayared structures. The CNM formed were 30-2500 nm in size.

Keywords: Carbon Nano Material (CNM), oil rich non-edible seeds, Taguchi optimization, S/N ratio, Scanning electron microscopy (SEM)

I. INTRODUCTION

The discovery of carbon nano materials (CNM)[1], its unique structural, chemical, mechanical and electrical properties and various possible applications has prompted scientists to focus their attention on this novel material[2]. Due to increasing demand of CNM for commercial purpose, efforts are being made to reduce the cost of its production. Chemical vapour deposition (CVD) or pyrolysis method for the synthesis of CNM is economically cheaper than the other methods. Petroleum-derivatives (e.g. benzene, acetylene etc,) are used for the synthesis of CNM which are expensive and non-renewable. Sharon's group have been first to initiate the research on the use of plant derived products like camphor, oil, non-edible plant parts, resin etc. as precursor for the synthesis of CNM[3-6] because plant tissues contain oil, carbohydrate, protein etc which are rich source of hydrocarbon. Literature survey indicates that there are many attempts to synthesis of CNM from plant derived and plant based precursors such as camphor[7], turpentine oil[8], bamboo[9], soyabeen seed and baggas[10], baggas, cashew nut and ritha seeds[11], tea leaves[12], cotton[13]etc.

In the present work, we have attempted to optimize the parameters and synthesis of CNM using oil rich non-edible seeds as precursor and single zone CVD set up for pyrolysis. For optimization of parameters Taguchi optimization method is used. We have selected three different oil rich non-edible seeds i.e. Ricinus communis, Madhuca indica and Azadirachta indica; their common names are castor, mahua and neem respectively, as a precursor for the synthesis of CNM. These seeds contain different amount of oil, carbohydrate and protein constituents, these are rich source of carbon. Since earlier study, worked on different plant based precursors such as stems, leaves, fibers, latex, seeds etc. but no one worked on Taguchi Optimization and synthesis of oil rich non-edible seeds. Hence, in this present work, we have optimized parameters by this statistical method and CNM synthesized using CVD method. The advantage of the Taguchi optimization method is to reduce numbers of experiment into few experiments and optimized parameters for effective results. So due to this, we used Taguchi Optimization for this present work.

II. MATERIALS AND METHODS

For synthesis of Carbon nano materials (CNM) using three different non-edible oil rich seeds Ricinus communis (Castor), Madhuca longifolia (Mahua) and Azadirachta indica (Neem) were used as natural precursors and procured from local market. For CVD process, the selected seeds were treated for removal of seed coat followed by drying. Castor seeds – Ricinus Communis L. (Euphorbiaceae), Mahua – Madhuca longifolia (Koening) Macbride (Sapotaceae), Neem – Azadirachta indica A. Juss. (Meliaceae), were soaked in 2% alkali (NaOH) for varying periods depending upon the hardness of seed coats[14]. Soaking was made till the imbibitions took place and thereby, seed coats were loosened. The seeds were removed from alkali and washed under running tap water. Subsequently washed seeds were rinsed with detergent followed by distilled water till the traces of alkali/ detergent washed off. The seed coat of R. communis, A. indica, M. longifolia were removed manually and finally cotyledons were collected for further investigations. The cotyledons were subjected to drying in muffle furnace at 60°C for 8-10 hrs[15]. The dried seed kernels were weighed and crushed in mortar pestle. 5 gm sample amounts of cotyledons were taken in quartz boat for further pyrolytic carbonization processes[13].

Chemical vapour deposition (CVD) set-up

Single zone CVD apparatus was used for the synthesis of carbon nano and micro materials from selected three oil rich non-edible seeds. The schematic of CVD set-up is presented in figure -1. 5 gm of crushed Seeds were taken in a quartz boat and placed in the center of 1 m long quartz tube (inside furnace) that was inserted in the furnace. To make the furnace oxygen free three different gases were used i.e. Argon (Ar), Nitrogen (N_2) and Hydrogen (H_2). Pyrolysis was done at three different temperatures i.e. 800^0C, 900^0C and 1000^0C for 1, 2 or 3 hrs. The carrier gas was flushed into the quartz tube at a higher flow rate (150 ml/min) so as to remove oxygen and the quartz tube was closed. Then the purging of gas was continued at the flow rate (25 ml/min) during pyrolysis. Desired temperatures was set when the temperature reaches 450^0C - 500^0C, the biomass starts vaporizing inside the tube and flows along with the carrier gas toward outlet of the tube. By the time the temperature reached 700^0C the flow rate was gradually decreased and stopped. When it reached at set temperature, it was maintained at that temperature for desired set duration. At high temperature, the hydrocarbons are decomposed and converted into simpler form of carbon. The non-carbonaceous material gets removed along with the carrier gas from the furnace and remnant carbonized carbon material are left in the quartz boat. Once the pyrolytic reaction was over the furnace was allowed to cool down to room temperature. The pyrolyzed carbon material was collected from the boat. To know the impact of parameters on the yield of CNM, factor effect were calculated. Details of methodology for the calculations of Taguchi Optimization technique are also discussed.

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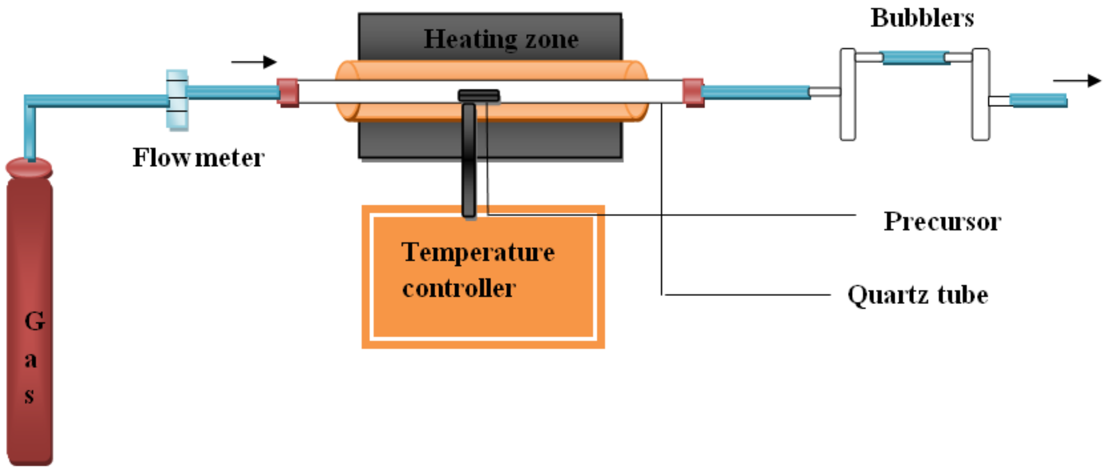

Figure 1: Schematic of a pyrolysis set-up using single zone CVD furnace

Standardization of synthesis parameters (Taguchi Optimization of Parameters)

For the present CVD process four parameters, each with three variables were to be tried. This would mean a minimum of 81 set of experiments and if it is done in triplicate it would be running 243 pyrolysis experiments to be carried out. Therefore, for the present work, experiment was designed by Taguchi Optimization method. This method is a statistical method developed by Genichi Taghuchi (1950) of Nippon Telephones and Telegraph Company, Japan for improving the manufactured goods, marketing & advertising, more recently, even applicable for the research field. In this method statistically designed orthogonal arrays[16 & 17] are being of used to evaluate comparatively smaller number of experiments. It is a simple efficient and systematic approach for the optimization of experimental parameters[18 & 19]. Using Taguchi method one can get information about the most effective parameters controlling the formation of a required product by carrying out only 9 experiments. The Taguchi philosophy of variability reduction is based on the fact that there is a 'best' value for the product. The Taguchi strategy attempts to find the combination of the values of the controllable design variables that minimizes the expected loss over the uncontrollable noise space. This experimental design provides a simple efficient and systematic approach for the optimization of experimental designs for performances, quality and cost[20].

Application of Taguchi Optimization for Present work

Four parameters considered for the synthesis of CNM from plant seeds are Carrier Gases, Precursors, Temperatures, and Duration of Pyrolysis. In total, three levels of each parameter were selected (Table I).

Table I Four parameters used for Taguchi Optimization Technique for oil-rich non- edible seeds

SN	PARAMETERS	LEVELS OF PARAMETERS		
		LEVEL 1	LEVEL 2	LEVEL 3
1	GAS	Ar	N_2	H_2
2	TEMPERATURE(^{0}C)	800	900	1000
3	DURATION(hr)	1	2	3
4	PRECURSOR(Seeds)	Ricinus communis	Azadirachta Indica	Madhuca Longifolia

Using above mentioned parameters and its level, L9 orthogonal matrix was designed (Table I). Taguchi optimization method is a statistical method[21] in which simple mean of analysis and optimization of complex systems based on the statistical analysis of data are optimized. To study the entire parameter space with only a small number of experiments orthogonal arrays (OA) is designed by using Taguchi optimization method. This approach of the statistical analysis is comparatively simple for optimization of experimental designs in order to evaluate performance and quality of the experiments.

Table II L9 orthogonal array of parameters as per Taguchi Optimization Technique for oil rich non-edible seeds

Taguchi Level	Gas	Temperature (^{0}C)	Duration (hr)	Precursor (Seed)
L1	Ar	800	1	Ricinus communis
L2	Ar	900	2	Madhuca longifolia
L3	Ar	1000	3	Azadirachta indica
L4	N_2	800	2	Azadirachta indica
L5	N_2	900	3	Ricinus communis
L6	N_2	1000	1	Madhuca longifolia
L7	H_2	800	3	Madhuca longifolia
L8	H_2	900	1	Azadirachta indica
L9	H_2	1000	2	Ricinus communis

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Statistical analysis of signal to noise (S/N) ratio and an analysis of variance (ANOVA) [22] has to be employed for determining relative importance of various factors. Orthogonal array (OA) analysis is completed by ANOVA and S/N ratio. ANOVA is used to analyze the results of the OA (Orthogonal Arrays) experiment and to determine how much variation each quality (influencing factor) has contributed.

S/N ratio is log function of desired output for optimization, which thereby, helps in data analysis and as well as in prediction of optimal results. An S/N ratio is a key part of the Taguchi optimization method and is often evaluated using a parameter design. Taguchi's S/N ratios are the performance statistics that recommends for selecting the best combination of the design variables. Larger the S/N ratio more robust the design. For the best design, the parameter which gives S/N ratio highest value is considered as the favoured parameter. Thus, to minimize the expected loss, one always finds the combination of the design variables, which maximizes the S/N ratio performance statistic.

The S/N is the ratio of the average response of the root mean square variation about the average response. Larger the S/N ratio, smaller the measurement of error because the S/N ratio is the reciprocal of the variance of the measurement error. S/N ratio is used to analyze the test run results because the S/N ratio signifies the mean and variation (scatter) of the experimental results.

Calculations for 'Larger the Better': There are three categories of S/N ratios (i) smaller the better, (ii) larger the better and (iii) nominal the best. In the present work target was to get larger quantity of carbon, therefore, "larger the better" is used for calculation using following equation:

$S/N = -10\log_{10} [(1/n)\ \Sigma 1/y_i^2]$ ------------------ (1)

Where **"y_i"** is the mean response calculated as y =1/n Σy_i and n is the number of experiments carried out under similar conditions.

Calculations for Effect of each parameter: To determine the effect of each parameter level (m_i) average value of S/N ratios was calculated for each parameter, using analysis of mean (ANOM). For this calculation, the S/N ratios of each experiment with corresponding parameter levels are calculated using following equation:

$m_i = (1/n_i)\ \Sigma S/N$ --------------------- (2)

Where, n_i is the number of experiments repeated with the same parameter levels.

There are two types of average value of S/N ratio are calculated. One is the overall mean S/N ratio calculated from the entire experiments for example from L9 experiment of L9 orthogonal array (i.e. from S/N values given in table: 3). Secondly, average S/N ratio is calculated for each parameter from equation-1. The advantage of the average value of S/N ratio, are considered to be the least effective parameters as compared to those parameters whose S/N ratios are larger than mean S/N ratio.

The parameters effects, i.e. the contribution of each experimental parameter to the quality characteristic are calculated by the analysis of variance (ANOVA). ANOVA is completed by SOS (summing of square) of variance of all levels of a given parameters. The relations that are used to determine the sum of the squares and the factor effect are given by the following equation:

$(SOS) = \Sigma N_i\ (m_i - \langle m_i \rangle)^2$ ----------------------------- (3)

Where m_i gives the average of the levels contributions for each parameters levels to S/N ratio, $\langle m_i \rangle$ is the average of mi for a given parameter and the coefficient and Ni denotes the number of repeats the experiment is conducted with the same factor level.

Summing of square (SOS) is standarized with respect to the degree of freedom (DOF) of the corresponding process parameters.

DOF = number of parameter level – 1 -------------------- (4)

If there are three levels for each parameters then DOF = 3-1 = 2

Some of square (SOS) of variances for all levels for a given parameter are divided by degree of freedom (DOF) of corresponding process parameter to obtain factor of effects (FOE) of various experimental parameters. Calculation of factor of effect (FOE) is calculated by the following equation:

FOE = SOS/[DOF x (SOS/DOF)] -------------------------- (5)

Finally, percentage parameter effect is calculated as following:

Parameter effect % = 100 x FOE --------------------------- (6)

Using L9 orthogonal array nine sets of experiments were carried out. Results were calculated based on equations 1 to 3 mentioned above, nano and micro carbon materials were synthesized by CVD method from precursors.

Pyrolysis Parameters and Their Levels for L-9 Orthogonal Array

According to Taguchi methodology, table was constructed from different combination of all the four possible parameters with a minimum of three variables of each parameter. Selected variable parameters (such as carrier gas, temperature, duration and different type of oil rich non- edible seeds) are orthogonal to each other. By using Taguchi Optimization technique, an orthogonal table was constructed in which three levels of a parameter was used. Therefore, L9 orthogonal table was constructed by using the parameters and their levels as mentioned in the table 1. Pyrolysis was carried out for each set of condition as mentioned in table II. Percentage (%) yield of CNM and its S/N ratio were calculated for each experimental result by using equation 1. The yields of CNM as well as S/N ratio calculated from each set are also given in table III.

Table III Orthogonal Array of parameters as per Taguchi Optimization technique, set for pyrolysis of Ricinus communis, Madhuca indica and Azadirachta indica seeds. Yield and S/N ratio of carbon CNM calculated as per Taguchi Larger the better calculations.

Taguchi level	Gas	Temperature (^0C)	Duration (hr)	Precursor (Seed)	% yield	S/N Ratio
L-1	Ar	800	1	R. communis	16.24	24.21
L-2	Ar	900	2	M. indica	12.12	21.67
L-3	Ar	1000	3	A. indica	13.70	22.73
L-4	N_2	800	2	A. indica	16.16	24.17
L-5	N_2	900	3	R. communis	14.16	23.02
L-6	N_2	1000	1	M. indica	11.46	21.18
L-7	H_2	800	3	M. indica	11.52	21.23
L-8	H_2	900	1	A. indica	15.64	23.88
L-9	H_2	1000	2	R. communis	14.10	22.98
				Mean S/N Ratio		22.78

Purification of carbon

Lumps of carbon collected from the quartz boat were powdered for acidic purification. To get rid of any metal or other residual material. Mixture of carbonized seeds and 6 M HCl [23] was thoroughly mixed and kept for 2 hrs and then filtered. The powder was then dried by keeping at 60^0C for 6 hrs and stored for further analysis.

The morphological observation of finally prepared CNM obtained from oil rich non-edible seeds as precursor was observed by Scanning Electron Microscope (SEM). SEM was examined by using 10-20 KeV with Hitachi 8-500 SEM and photographed with Kodak Tmax 100 film.

III. RESULTS AND DISCUSSION

Impact of pyrolysis on the synthesis of CNM

Pyrolysis is one form of energy recovery process, which has the potential to generate carbonaceous material, oil and gas product[24]. Because of the thermal treatment, which removes the moisture and volatile matter contents the biomass[13], the remaining solid carbonaceous material shows different properties than the parent biomass materials. The remarkable differences are mainly in their shape, size, porosity, surface area, pore structures (nano-porous, micro-porous, meso-porous and macro-porous) and physiochemical properties such as composition, elemental analysis and ash content[25]. Pyrolysis temperature has the most significant effect-followed by pyrolysis heating rate, the carrier gas flow rate and then finally the pyrolysis residence time[24]. An increased pyrolysis temperature (800-1000^{0}C) leads to a reduced yield of solid carbonized materials and increased the yield of liquid and gases. As the temperature is raised, there is a rise in fixed carbon percentage and there is a decrease in volatile matter i.e., it might be due to greater primary decomposition of biomass at higher temperature or through secondary decomposition of carbonaceous materials residues. Indeed, as the temperatures of primary degradation are increased or the residence times of primary vapours inside the cracked particle have to stay shorter, the CNM yields decrease. In Taguchi optimization, the deviation S/N ratio shows at lower temperature (800^{0}C) and lower time of pyrolysis gives the high yield of CNM. But in the case of carrier gases, Argon is the best parameter rather than nitrogen and hydrogen for the yield of CNM; hydrogen is a reducing gas so it seems that it reduced secondary decomposition of carbonaceous materials residues and affects the yield of CNM.

The formation of CNM from oil rich non-edible seeds consist pyrolysis using Taguchi optimization method to break down the cross-linkage between carbon atoms into nano size carbon materials. The pyrolysis of biomass is a complex phenomenon that is difficult to discover kinetic models that explain the mechanism of thermal decomposition. In many of kinetic formulation of solid state reactions, it has been assumed that the isothermal homogeneous gas or liquid phase kinetic equation can be applied.

Impact of parameters as Taguchi optimization on the yield of carbon

So far as yield of carbon from pyrolysis of different seeds are concerned R. communis and A. indica yielded 14 – 16% carbon, whereas M. indica produced 12% carbon. Various parameters of pyrolysis do not seem to have much impact on the yield. However, a trend in case of all the 3 seeds was observed i.e. increase in pyrolysis temperature leads to a reduced yield of solid carbonized materials and increased the yield of liquid and gases. This decrease in CNM yield with increasing temperature could either be due to greater primary decomposition of biomass at higher temperature or through secondary decomposition of carbonaceous materials residues. Signal to noise (S/N) ratio of CNM produced under different conditions is plotted in figure 2. The purpose of this work was to find out the best set of parameters for pyrolysis which could give the maximum amount of CNM. "Larger the Better" condition was used to calculate the S/N ratio was more positive to the mean value of S/N ratio were considered as suitable levels of parameters for which S/N ratio were considered as suitable levels for the production of CNM (figure 2). Among selected three types of oil rich non-edible seeds A. indica seeds appeared to give the best yield.

In Taguchi optimization, the deviation S/N ratio shows that lower tried temperature (800^{0}C) and lower time of pyrolysis (1hr) gives the high yield of CNM.

Amongst the three tried carrier gases, Argon is the better suited than nitrogen and hydrogen for the yield of CNM. Since hydrogen is a reducing gas it might have reduced the carbon during secondary decomposition of carbonaceous materials

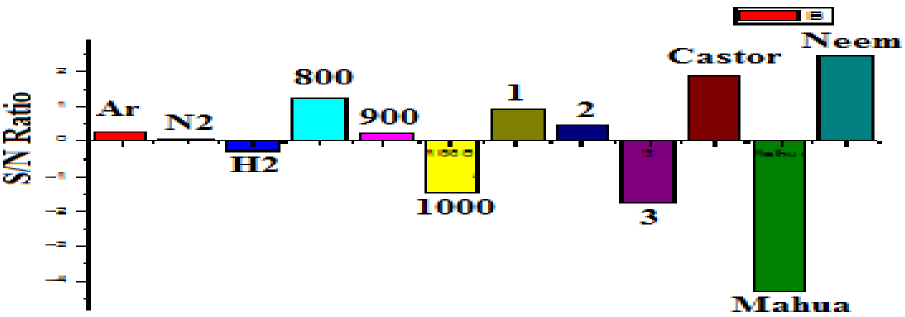

Fig. 2 Schematic shows the impact of parameter on the CNM synthesis from oil rich non-edible seeds (R. communis , M. indica, A. indica), as per Taguchi calculations of Deviation S/N ratio

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The calculation of percentage impact of each parameter on the production of CNM (Table IV) suggests that precursor (80.30%) has maximum impact on the experimental designing followed by temperature (10.85%) then duration (8.50%) and the least impact is that of carrier gas (0.35%) which was shown in the pie diagram (figure 3).

Table IV Control parameters of the Pyrolysis of non-edible seeds (R. communis, M. indica and A. indica) and corresponding S/N ratio.

Expt. No	**Gas**			**Temperature (^{0}C)**			**Time duration (hr)**			**Precursor (seed)**		
	Ar	**N_2**	**H_2**	**800**	**900**	**1000**	**1**	**2**	**3**	**R. communis**	**M. indica**	**A. indica**
L1	24.21			24.21			24.21			24.21		
L2	21.67				21.67			21.67			21.67	
L3	22.73					22.73			22.73			22.73
L4		24.17		24.17				24.17				24.17
L5		23.02			23.02				23.02	23.02		
L6		21.18				21.18	21.18				21.18	
L7			21.23	21.23					21.23		21.23	
L8			23.88		23.88		23.88					23.88
L9			22.98			22.98		22.98		22.98		
Sum (S/N)	68.61	68.37	68.09	69.61	68.57	66.89	69.27	68.82	66.98	70.21	64.08	70.78
Deviation S/N	0.25	0.01	-0.27	1.25	0.21	-1.47	0.91	0.46	-1.78	1.85	-4.28	2.42

m_i	22.87	22.79	22.70	23.20	22.86	22.30	23.09	22.94	22.33	23.40	21.36	23.59	
<m_i>	22.78			22.78			22.78			22.78			
m_i - <m_i>	0.09	0.01	-0.08	0.42	0.08	-0.48	0.31	0.16	-0.45	0.62		-1.42	0.81
SOS	0.04			1.24			0.97			9.17			
SOS /DOF	0.02			0.62			0.485			4.585			
FOE (%)	0.35			10.85			8.50			80.30			

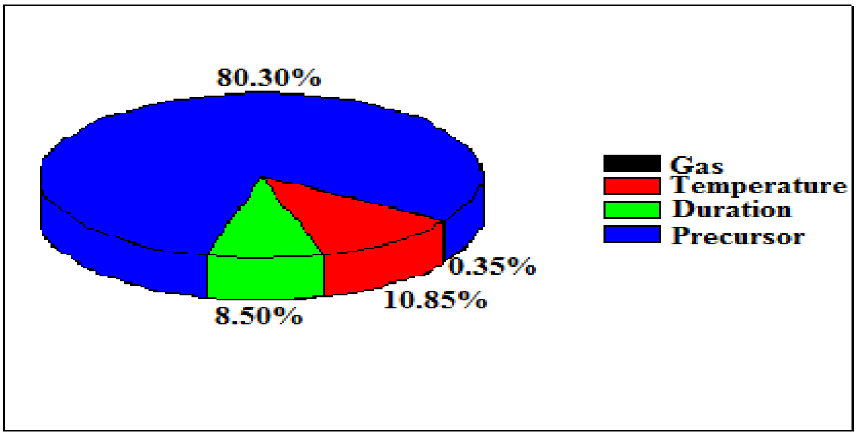
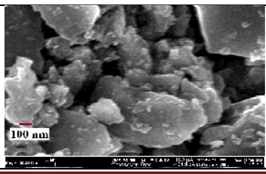


Fig. 3 Schematic of Pie chart of (%) factor of effects of each parameter

Morphology of carbon nano materials produced by pyrolysis

Morphological structures and observations of SEM micrograph of pyrolised oil rich non-edible seeds (Ricinus cummunis, Madhuca longifolia and Azadirachta indica.) are represented in the table-V.

Table V: SEM micrographs of oil rich seeds (Ricinus communis seed, Madhuca longifolia seed and Azadirachta indica) in relation to different parameters (carrier gases, different temperatures & duration) with their corresponding CNM.

Seed	**Parameters Gas–Temp^{0}C-Duration Hr-Precursor**	**SEM Images**	**CNM Morphology (SEM)**
Ricinus communis	L1 Ricinus communis seed (Ar, 800^{0}C, 1 hr)		Smooth surface plates with small fragments.

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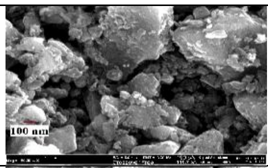
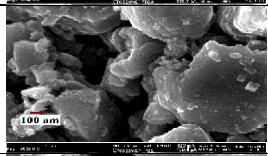
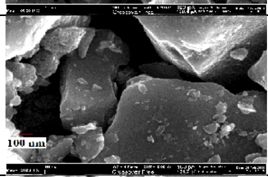
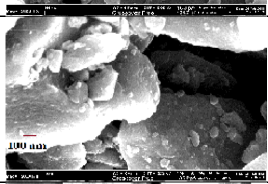
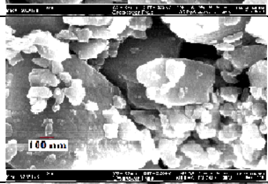
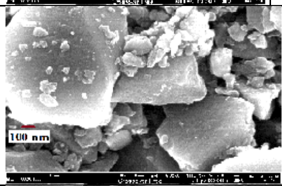
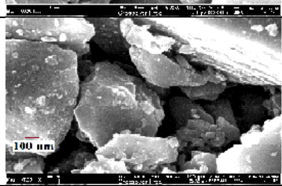
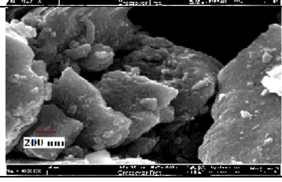
	L5 Ricinus communis seed (N_2, 900^{0}C, 3 hrs)		Smooth surface plates with small fragments. Thickness of 100mm plate
	L9 Ricinus communis seed (H_2, 1000^{0}C, 2 hrs)		Smooth surface plates with small fragments.
Madhuca longifolia	L2 Madhuca lonfigolia seed (Ar, 900^{0}C, 2 hrs)		Rough surface plates with small fragments and in two SEM side of plate show porous structure.
	L6 Madhuca longifolia seed (N_2, 1000^{0}C, 1 hr)		Rough and rippled surface plates with small fragments.
	L7 Madhuca longifolia seed (H_2, 800^{0}C, 3 hrs)		Rough surface plates with small fragments.
Azadiracht a indica	L3 Azadirachta indica seeds (Ar, 1000^{0}C, 3 hrs)		Plate with rippled surface and sides showing many layers of plates also small fragments.
	L4 Azadirachta indica seeds (N_2, 800^{0}C, 2 hrs)		Plate with rippled surface and sides showing many layers of plates also small fragments.
	L8 Azadirachta indica seeds (H_2, 900^{0}C, 1 hr)		Plate with rippled surface and sides showing many layers of plates also small fragments.

The SEM micrographs of CNMs (synthesized through CVD of three precursors (oil rich non-edible seeds: Ricinus communis, Madhuca longifolia and Azadirachta indica) are shown in this table. SEM micrographs are of the experiments for different levels (L1 – L9) as optimized by 'Taguchi method'. The SEM micrograph were obtained from the powder of precursors which revealed plate like structures. In L-series (Table-5), the SEM micrograph of pyrolysed carbon nano materials (CNM) of Ricinus communis seeds L1, L5 and L9 showed larger sized structure (100-2500 nm) with smaller fragments (30-100 nm). The morphology of CNM was found smooth plate like structures. In another investigation[26] had synthesized chain of carbon nano beads from castor seeds as precursor, however, utilizing Argon gas at 900^{0}C with Iron as catalyst. The SEM micrograph of CNM of Madhuca longifolia seeds L2, L6 and L7 exhibited about 250-2000 nm for large size structure and the smaller fragments were in the range of 40–200 nm. The morphology of CNM was rough surface plate like structure but in L2, two sides of plate structure showed porous structure. In L6, the surfaces of

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CNM were rough as well rippled surface plate. Earlier, Viswanathan et al. (2014)[27] found plate of carbon with holes by observing the SEM micrograph of CNM obtained from Calotropis sap (at 900^{0}C, argon as carrier gas and nickel as catalyst for 2 hours).High resonance (SEM) micrograph of Azadirachta indica seeds L3, L4 and L8 as observed had the size of large structure (250-2500 nm) and the size of smaller fragments (40-200 nm), the morphology of CNM showing plate with rippled surface and side showing many layers of plates. Porous carbon materials from Neem seeds (treated with H_3PO_4) were observed by Kenneth et al. (2015)[28].

IV. CONCLUSIONS

Carbon nano materials (CNM) synthesized from biological precursors oil rich non-edible seeds (R. communis, M. indica and A. indica seed) have been synthesized by pyrolysis method using single zone CVD furnace without using metal catalyst. The process parameters have been optimized for the bulk growth of CNM from these seeds by Taguchi optimization method in which controlling parameters are oil rich non-edible seeds (R. communis, M. indica and A. indica) as a precursors, temperature (800^{0}C, 900^{0}C and 1000^{0}C), carrier gas (Ar, N_2 and H_2) and duration of pyrolysis (1 hr, 2 hrs and 3 Hrs). Signal to noise (S/N) ratio was evaluated in order to find out the best set of parameters for pyrolysis, which could give the maximum amount of CNM. The condition “Larger the Better” was used to calculate the S/N ratio for the production of CNM. Among three types of seeds Azadirachta indica seeds appeared to give the best yield, followed by castor seeds.in Taguchi optimization, the deviation of S/N ratio showed that the lower tried temperature (800^{0}C) and lower time of pyrolysis (1 hr) gives the higher yield of CNM. While comparing different carrier gases, it was observed that Argon better suited than Nitrogen and Hydrogen for the yield of CNM. The calculation of percentage impact of each parameter on the production of CNM suggested that precursor (80.30%) had maximum impact on the experimental designing followed by temperature (10.85%) and duration of pyrolysis (8.50%), however, carrier gas had least impact (0.35%). SEM characterization of synthesized CNM shows plates like morphology and size varied from 30-2500 nm in range.

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