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Optimization of Synthesis Parameters for Multiwall Carbon Nano Tubes and its Electrical Application.

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Abstract:

Chemical Vapour deposition (CVD) is most utilized method to synthesize Multiwall Carbon Nano Tubes (MWCNTs) because of simple, economical, easy to control experiment condition, controllable diameters and better yield compare to others methods. Plant-based precursors i.e., Olive oil was used as source of carbon to obtained MWCNTs over Ni-Co catalyst in inert atmosphere of Argon gas. The effect of synthesis parameters on yield and morphology of MWCNTs were evaluated using Taguchi optimization method. Acid treated MWCNTs were characterized using FT-IR, FEG-SEM, EDS, TEM. The average diameter and length of MWCNTs are 48 nm and 736 nm, respectively. The obtained electrical conductivity was 0.477 S/cm using Four-Probe Method.

Keywords:

Chemical Vapour deposition, Olive oil, Plant-based precursors, FT-IR, FEG-SEM, EDS, TEM

Introduction:

Carbon Nano Tubes (CNTs) were discovered in 1991 as Multiwall Carbon Nanotubes (MWCNTs) by Sumio Iijima (Iijima & Ichihashi, 1993). Soon thereafter Multiwall Carbon Nano Tubes (MWCNTs) have been led to intense interest in nanomaterials research. These nanometer-size materials are a one-atom thick sheet of as graphite sheets rolled into cylindrical tubes with a diameter of nanometer scale. Extensive research on carbon nanotubes leads its mechanical, thermal, and electrical properties, which revealed them to be materials with great potential for both fundamental research and various applications such as Microwave absorbing materials(Micheli et al., 2010), sensors (Monereo Cuscó, 2016), hydrogen storage (Jaybhaye et al., 2007), electronics (Nasir et al., 2018), and catalysis (Gulino et al., 2005).

Conventionally, synthesis of Carbon Nano materials (CNMs) like MWCNTs and Carbon Nano fibre's (CNFs) is carried using variety of different techniques (T. Singh et al., 2018), and phase of precursors which are non-renewable origin and are derivatives of petroleum-based hydrocarbons and coal (Kshirsagar et al., 2006) Till date mostly purified petroleum products such as methane, ethane, benzene, xylene are popular choices for synthesizing CNMs (Tibbetts et al., 2006). The factor which affects the type, yield and quality of CNMs are precursor, catalyst, temperature, carrier gas and reaction times. Thus, optimization of the control parameters for a desired form, yield and quality of the CNMs is investigated by many researchers.

Renewable plant-based precursors have the potential to address the above problems. The use of locally available plant-based precursors like oil are the better choices for the synthesis of MWCNTs. Use of the renewable plant-based precursors makes the process green as it conforms to the principle of green chemistry and green technology. Thus, it is very important to explore new renewable precursors for the synthesis of MWCNTs.

To study the synthesis parameters variable and its effect on synthesis process using traditional experimental techniques involves large numbers of experiment and inefficiency in relationship between chemical and physical parameters, experimental design which effect the cost of the final product (Shafiee et al., 2019).

In the recent study, in order to achieve good yield, control of morphology, determination of the effect of synthesis parameters and their quantitative limited range, detailed design of experimental conditions is needed. One of the best design experimental methods is Taguchi method. Taguchi methods are statistical methods developed by Genichi Taguchi to improve the quality of manufactured goods and more recently also applied to engineering (Taguchi & Phadke, 1984)(Boucher, 2011).This method allows for the analysis of many different parameters without an excessively high amount of experimentation (Cobb & Ciarkson, 1994; Pignatiello, 1988). In this way, it allows for the identification of key parameters that have the most effect on the characteristic value so that further experimentation on these parameters can be performed and the parameters that have little effect can be ignored (Cobb & Ciarkson, 1994; Popoola et al., 2020). The steps involved in this method are (1) screening the parameters, that effect on reaction process variables and (ii) identification of major factors. Taguchi method based on orthogonal array (OA) for Design of experiment, contains the variable parameters and level involves in reaction process (Mahto & Kumar, 2008; A. Singh et al., 2020).

Recently, the Taguchi method has attracted the attention of researchers for optimizing Chemical methods (Anand & Srivastava, 2015), physical methods (Kosobudskii et al., 2018)], biochemical and biological methods (Zeraati et al., 2021), in order to ensure acceptable results and also used in various industries processes, production and quality control.

In this research article we report a novel, environmental friendly, cost efficient, low waste production and green approach for synthesis of MWCNTs and its parameters were optimized using Taguchi method. Synthesis parameters were optimized i.e., Catalyst, Temperature and Reaction annealing time.

Experimental:

Materials:

All chemical used for synthesis of CNTs are of Analytical grade. Olive oil punched from Ashwin Fine Chemical and pharmaceuticals, Thane. Hydrogen and Argon gas was supplied from gas ward, Ulhasnagar.

Methods:

Synthesis of MWCNTs:

The growth of MWCNTs was obtained by Chemical Vapor Deposition (CVD) of Olive oil as Carbon source over three different catalysts like Ni, Co and Ni-Co NPs. A quartz tube (D) (1 meter in long and 1.5 cm in diameter) is located inside the furnace such that approximately half the tube is covered with vaporizing furnace (B) and pyrolysing furnace (C). A quartz boat containing the oil is place at the center of vaporizing furnace. About known quantity of the catalyst powder dispersed into the center of Quartz tube (B) at pyrolysing furnace (C). After closing the CVD Furnace, hydrogen gas was purged into quartz tubes to remove Oxygen.

The pyrolysing furnace (C) was heated at set temperature (700^{o}C, 800^{o}C and 900^{o}C) to anneal the catalyst. The vaporizing furnace was heated and the vapors of oil were carried into pyrolyzing furnace over the catalyst with the help of hydrogen gas and reactor cools down to room temperature (Ramakrishnan et al., 2014). The schematic diagram of CVD setup used for the synthesis of CNTs using Oil are shown in the Figure 1.

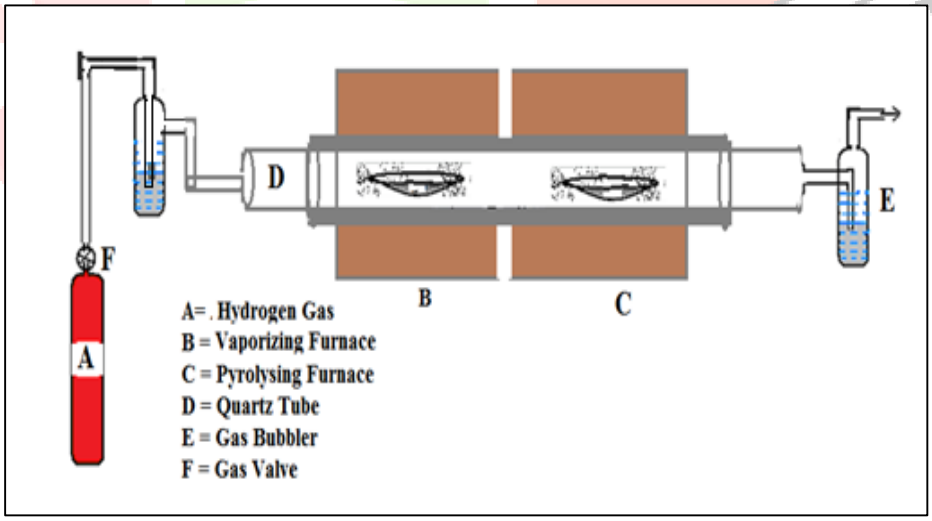

Figure 1 Schematic diagram of CVD setup for the synthesis of MWCNTs using olive oil

Optimization of Synthesis Parameters

The major concepts in Taguchi experimental design are; factors, levels, orthogonal array and optimum condition. Factors are the variable which affecting the experimental response, level are the number of factors present in the experiments and O.A. are actual setup of experiments. There are different O.A. set according to condition, for example, L9 array is most commonly used for 3 factors each in 3 levels.

Parameters like Temperature, Catalyst and Reaction time were optimized using this technique for synthesis of MWCNTs shown in Table 1.

Table 1. Parameters, Levels use in Taguchi optimization technique for synthesis of MWCNTs

Parameters		Level		
		1	2	3
A	Temperature (^oC)	700	800	900
B	Nano Metal Catalyst (100 mg.)	Ni	Co	Ni-Co
C	Reaction Annealing Time (min.)	60	90	120

Design of orthogonal arrays and L9 experiments:

The orthogonal experiment design was selected to investigate the effect of various parameters and obtained optimal as well as well-functioning procedure for synthesis of MWCNTs. Various synthesis parameters i.e., Temperature, Catalyst and Reaction annealing time were optimized using Taguchi optimization technique shown in table 2.

Table 2. Orthogonal array and L9 experiment used in synthesis of MWCNTs using oil

Level	Temperature (^{o} C)	Catalyst (NPs)	Annealing time (min.)
L1	700	Nickel	60
L2	700	Cobalt	90
L3	700	Nickel-Cobalt	120
L4	800	Nickel	120
L5	800	Cobalt	60
L6	800	Nickel-Cobalt	90
L7	900	Nickel	90
L8	900	Cobalt	120
L9	900	Nickel-Cobalt	60

Purification of MWCNTs

As grown MWCNTs have impurities of catalyst particles and amorphous carbon. For purification purposes it is shocked in 50 % HCL, sonicated for 60 min and keep for 12hrs. After 12 h. washed it with distilled water, then above powder was shocked in 50 % HNO_3 for 12hrs, after that it was filter using whatman filter paper 41, and. washing it with distilled water till achieve the neutral pH. The black powder putted in to the oven at 150°C for drying. The final product i.e. purified MWCNTs was now used for characterization (Gaud et al., 2018; Jaybhaye et al., 2006).

Characterization

Purified MWCNTs powder obtained at optimal condition were characterized using analytical instruments such as powder Fourier Transform Infra-Red (FT-IR) Spectroscopy, X- ray Diffraction (XRD), Raman Spectroscopy, Field Electron Gun Scanning Electron Microscope (FEG-SEM), Energy Dispersive X-ray Spectroscopy (EDS), High Resolution-Transmission Electron Microscopy (HR-TEM) and Thermo-Gravimetric Analysis (TGA).

Results and Discussion:
Synthesis of MWCNTs from Olive oils:

The qualitative and quantitively synthesis of MWCNTs was easily carried out using 25 ml of Olive Oil over Ni, Co and Ni-Co catalysts. The nine experiments according to orthogonal array (L9) were conducted shown in table 2. The synthesis of MWCNTs was carried out using of Olive oil over different Catalyst (Ni, Co and Ni-Co), temperature (700, 800, 900^{o}C), with a typical reaction time of (60, 90, 120 min) for each deposition. All 9 sample synthesized at each level was purified and its result is mentioned in Table 3.

Table 3. Parameters and levels used for synthesis of MWCNTs using Olive Oil

Levels	Temperature (^{o}C)	Catalyst	Reaction Time (min)	Yield (g.) MWCNTs	S/N Ratio
O-1	700	Nickel	60	3.415	10.668
O-2	800	Cobalt	90	3.215	10.144
O-3	900	Nickel-Cobalt	120	4.100	12.256
O-4	700	Nickel	90	3.618	11.169
O-5	800	Cobalt	120	3.588	11.097
O-6	900	Nickel-Cobalt	60	3.576	11.068
O-7	700	Nickel	120	3.710	11.387
O-8	800	Cobalt	60	3.871	11.756
O-9	900	Nickel-Cobalt	90	3.581	11.080

S/N calculations

Orthogonal array experimental yield and S/N ratio of each experimental are shown in Table 3. From the yield, S/N ratio response was calculated by using the eq. larger the best (Srivastava et al., 2007; Taguchi & Phadke, 1984),

$$S/N = -10*\log \{\Sigma (1/n) *(1/Y)^{2}\}.$$

Where, y is the characteristic property and *n* is the replication number of the experiment. The obtained 9 samples as per as orthogonal arrays i.e., as grown MWCNTs and purified MWCNTs were characterized using XRD shown in figure 2. (a) and (b) respectively.

For as grown MWCNTs Compared with purified MWCNTs, the results indicate that as grown MWCNTs have a mixture of two phases: cubic crystal structure of metallic NPs and MWCNTs. The diffraction peaks at ~26°and ~43° can be well indexed as the (002) and (100) reflections of graphite Carbon structure of MWCNTs.

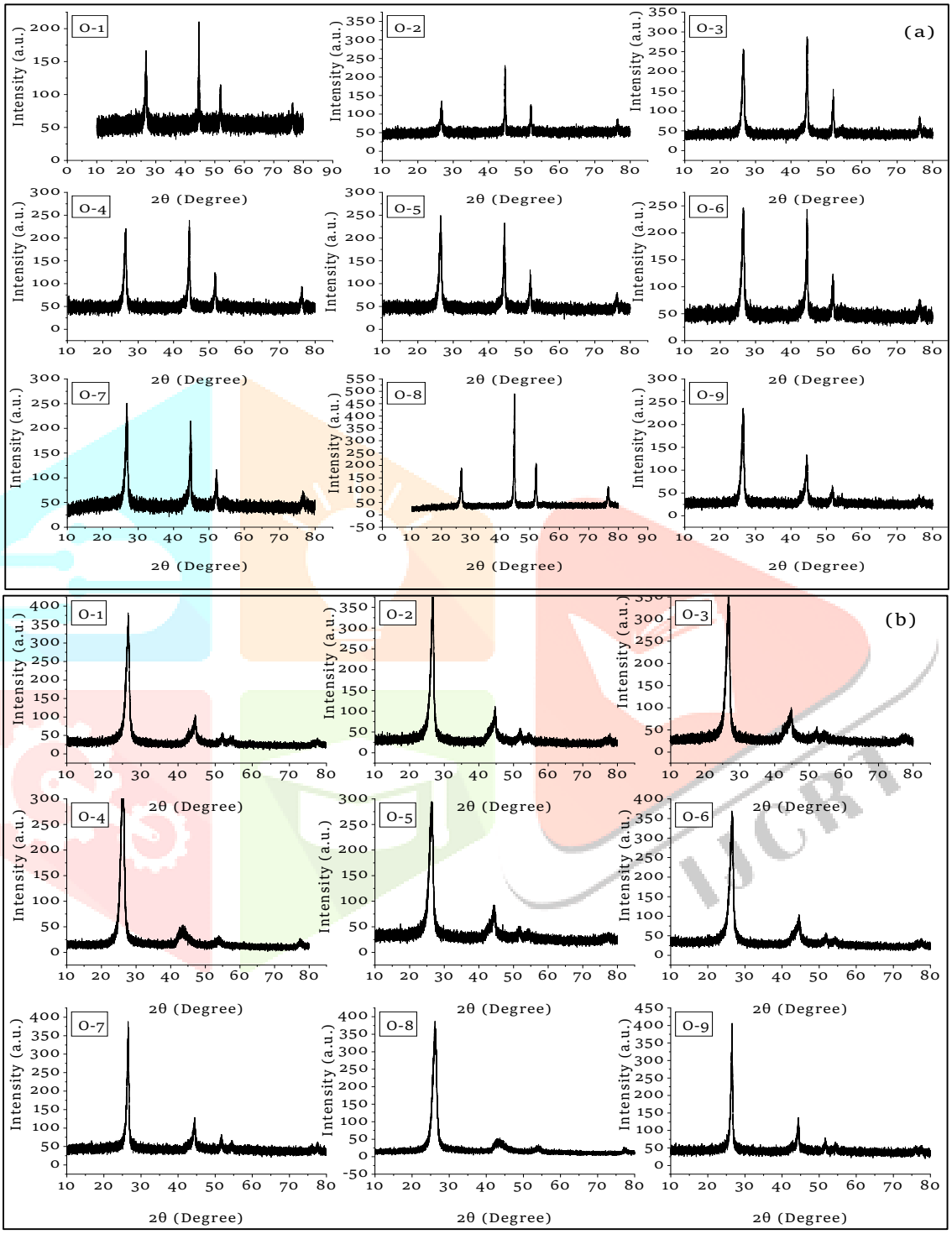

Figure 2. XRD pattern of
(a) As Grown MWCNTs and (b) Purified MWCNTs

Similar peak was observed in both as grown and purified MWCNTs of all 9 samples. In addition, the positions and relative intensities of peaks present in Figure 2 (a) (as grown MWCNTs), related with metallic NPs at $2\theta = \sim 45^0$, $\sim 53^0$, and $\sim 78^0$ indicate the presence of MNPs (used as catalyst) are disappears in Figure 2. (b) for purified MWCNTs; it can be concluded that catalyst is removed after purification.

Table 4. Control Parameters for synthesis of MWCNTs, Corresponding S/N ratio and FOE

LEVEL	Temperature (°C)			Catalyst			Reaction annealing time (min.)		
	700°C	800°C	900°C	Ni	Co	Ni-Co	60	90	120
O-1	10.668			10.668			10.668		
O-2	10.144				10.144			10.144	
O-3	12.256					12.256			12.256
O-4		11.169		11.169				11.169	
O-5		11.097			11.097				11.097
O-6		11.068				11.068	11.068		
O-7			11.387	11.387					11.387
O-8			11.756		11.756		11.756		
O-9			11.080			11.080		11.080	
Sum S/n	33.067	33.334	34.224	33.225	32.997	34.404	33.492	32.393	34.740
Deviation	-0.475	-0.207	0.682	-0.317	-0.545	0.862	-0.050	-1.149	1.198
mm_i	11.022	11.111	11.408	11.075	10.999	11.468	11.164	10.798	11.580
$< m_i >$	11.180			11.18			11.181		
SOS	0.244			0.380			0.920		
FOE(%)	15.841			24.614			59.545		

In addition to finding the condition for highest yield of MWCNTs, we also calculated the relative importance of each parameter. To find out the percentage influence of these levels and parameter on the synthesis and yield, Summing the Squares (SOS), Degree of freedom (DOF) and Factor of effects (FOE) were calculated by using equation

$$\text{SOS} = \sum_{i=1}^{i=j} N_i \{ m_i - < m_i > \}^2$$

Where,

$$mm_i = \left(\frac{1}{N_i}\right) \Sigma \frac{S}{N}$$

Gives the average of the level of contribution of each parameter to S/N ratio,

$< m_i >$ = is the average of m_i for a given parameter and

N_i - Represent the number of times the experiments are conducted with the same factor level.

$$\text{FOE} = \frac{SOS}{DOF \; X \; \sum \frac{SOS}{DOF}} * 100$$

Where, DOF is degree of freedom.

Figure 3 shows the FOE of each parameter. The figure 3 suggests that amongst all the three parameters, most significant parameters for synthesis and to obtained better yield for MWCNTs are annealing Time

(59%), whereas catalyst (25%) and temperature (16 %) have least and moderate significant on synthesis and yield.

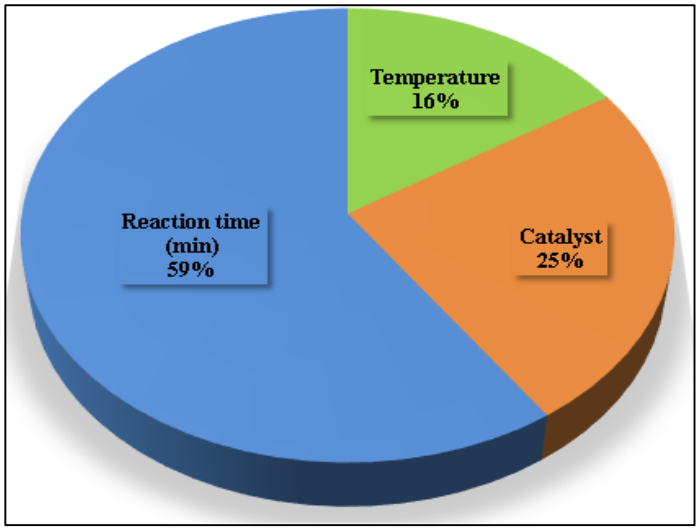

Figure 3 FOE of Effect of each parameter on yield of MWCNTs

However, these calculations show the % influence of parameters on the maximizing the yield but do not show the optimal condition for synthesis of MWCNTs. There for to find out the optimal conditions for yield the deviation of S/N ratio was calculated. The graphical representation of deviation of S/N ratio Vs synthesis parameters, was plotted, which are shown in Figure 4.

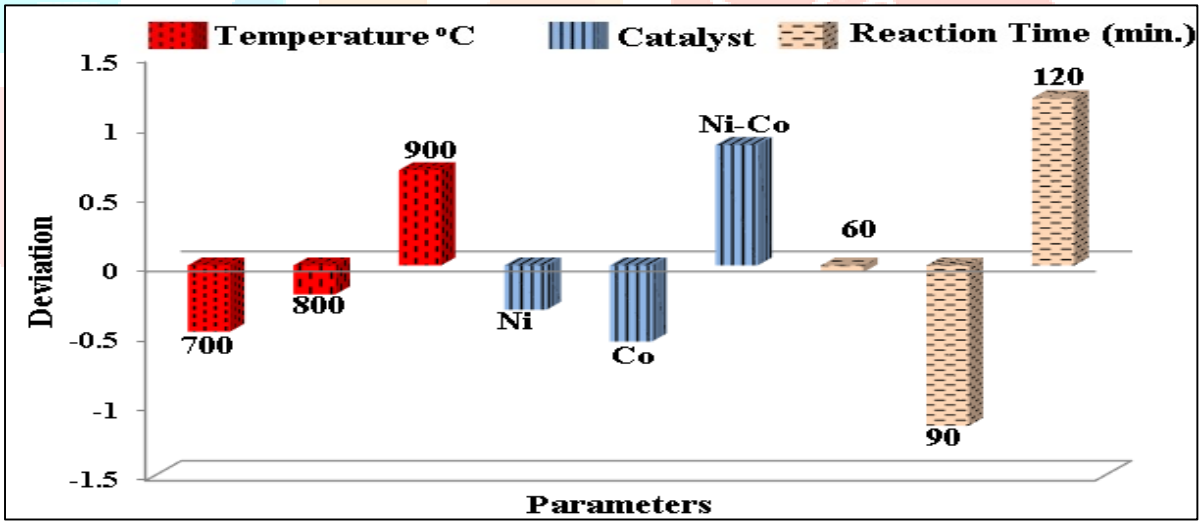

Figure 4 Deviation of S/N ratio vs. Parameters for synthesis of MWCNTs

Positive and negative effect of different levels on yield of the product can be obtained. Positive value of deviation of S/N ratio indicates dominance of the parameters on yield.

The greater is the S/N ratio, the smaller is the variance of yield around the desired value (Yiamsawas et al., 2011). Based on mean S/N ratio response as shown in figure 4 deviation of S/N ratio vs synthesis parameters graph, The best parameters for the synthesis of MWCNTs when olive oil pass over Ni-Co NPs at 900 ^{o} C temperature and annealed for 120 min. in inert atmosphere, it gives 50 %.

A new experiment, based on the optimal parameters obtained by Taguchi optimization technique was established. The MWCNTS was prepared using optimum condition and characterized. This study is

provided the better quality and control on yield of MWCNTS and synthesised MWCNTS by CVD method at optimal condition were characterized using FT-IR Analysis, XRD analysis. The morphology and purity of MWCNTS were analysed using SEM & EDX, HR-TEM and TGA.

XRD analysis:

XRD analysis of the as grown and purified MWCNTs samples was conducted and the results are shown in Figure 5. Figure 5. (a) shows the diffraction peak for as grown MWCNTs at around 45.62°,53.95° and 78.44° could be indexed as [101], [202] and [220] reflection for Ni-Co NPs as per the PDF JCPDS: 01-077-7529. Figure 5.(b) shows the XRD profiles of the purified MWCNTs and diffraction peak at around 2θ = 26.4° indexed as [002] reflection for graphitic carbon according to PDF JCPDS: 01-082-9929. The low intensity peak at 44^0 is attributed due to the graphite like structure corresponding to the [100] plane. The Figure 5. (b) revealed that only the characteristic graphitic peaks at 2θ values of 26.60^o, 44.0^o were left behind after purification. It was also confirmed that Ni-Co NPs was removed during the purification process. The lattice parameters obtained from JCPDS file of purified MWCNTs are presented in table 5.

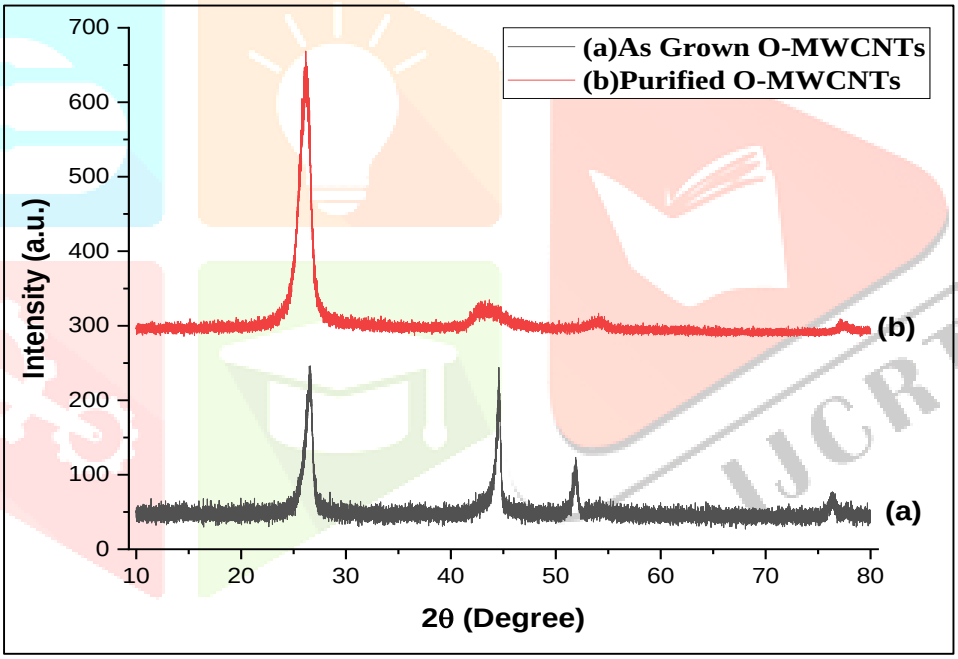

Figure 5. XRD image of MWCNTs synthesized at optimal condition (A) As Grown and (b) Purified MWCNTs

Table 5. lattice parameters and Porosity of MWCNTs

Sample	Lattice parameter			Cell volume (V)	Density		Porosity
MWCNTs	a (Å)	c (Å)	c/a	(Å)	XRD ($g./cm^3$)	Bulk ($g./cm^3$)	(%)
	2.466	6.727	2.727	35.45	2.26	1.10	48.67

The volume of unit cell was calculated by equation as in reference (Ullah et al., 2013).

$$\text{volume of unit cell (V)} = \sqrt{3}\ X\ a^2\ X\ \frac{c}{2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.1$$

$$V = 0.866\ a^2c$$

The porosity was calculated by equation as in reference (Ullah et al., 2013)

$$\text{Porosity (P)} = (1-\frac{\rho_b}{\rho_x}) \text{ X } 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.2$$

Where,

ρ_b = Bulk density of MWCNTs

ρ_x = X-ray density

Raman spectroscopy Analysis

Raman spectroscopy analysis supports the Purification and nature of CNTs synthesized by Olive Oil. Raman spectra of the purified CNTs via acid treatment were measured from 400 to 3500 cm^{-1} with lesser Raman (HR Raman) spectroscopy and shown in Figure 6. The Raman spectra of CNTs three well defined bands of carbon at 1342 cm^{-1}, 1580 $cm^{-1}$and 2674 cm^{-1} for D band, G band and 2D band respectively (Sharon & Sharon, 2006). The peak 1342 cm^{-1} is assigned to the D mode which corresponds to sp^2 hybridized carbon. The D band indicate disorders and defects present in MWCNTs as well as nature of the graphitic structure and G band is due to the C–C stretching in the graphitic material. The second order D-band (also called the 2D - band), which is related to the boundary point K in Brillouin zone of graphite and depends upon the packing in three-dimensional space, is evident around 2500–2900 cm^{-1} (Jaybhaye et al., 2007; Ramakrishnan et al., 2014). Moreover, the peaks at 2674 cm^{-1} are due to symmetric and asymmetrical C–H stretching vibrations of the CH_2 group. The intensity ratio of the D band to the G band (I_D/I_G) is often used to estimate the defect concentration in carbon materials. The low value of I_D/I_G ratio indicates high graphitization and high value of I_D/I_G ratio is usually attributed to the presence of defects on CNTs. The I_D/I_G ratio of MWCNTs is 1.20 indicates a high graphitization (Srivastava et al., 2007).

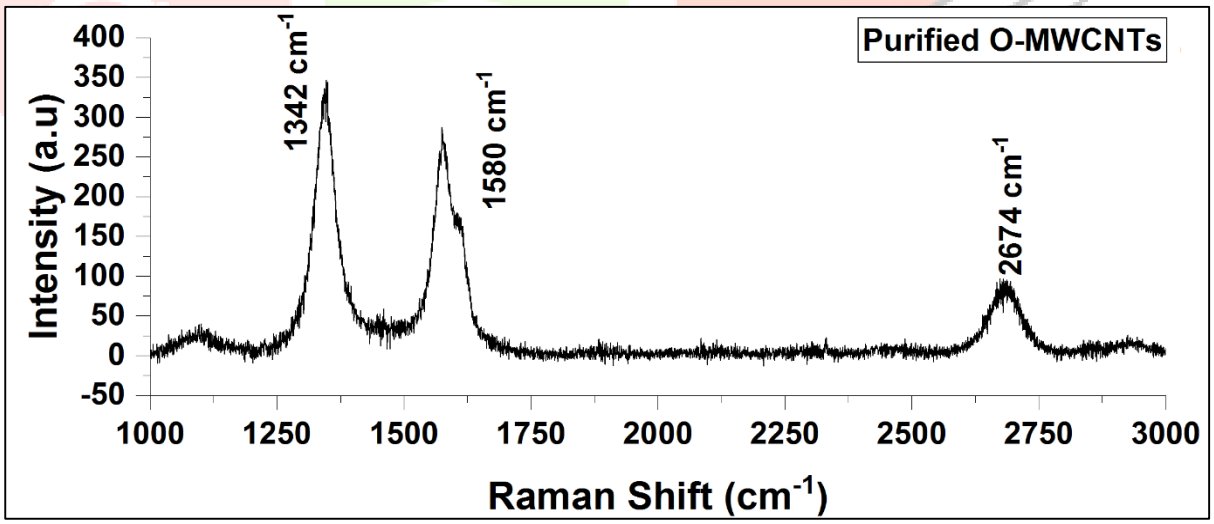

Figure 6. Raman spectra of MWCNTs synthesized at optimal condition

FEG-SEM Analysis:

The FEG-SEM was supports for morphologies of synthesized MWCNTs. The Figure 7. (a) and (b) shows the SEM images and EDX spectra respectively of purified MWCNTs, whereas figure 7. (c) and (d) shows tube diameter and tube length distribution respectively, of MWCNTs using SEM image, processing using the ImageJ software. The synthesized MWCNTs having diameter in the range of 30-70 nm and length ranging from 400-1400 nm. The average diameter and length of MWCNTs are 48 nm and 736 nm,

respectively. Figure 7 (b), Show the EDX spectra of purified MWCNTs, only one instance peak of carbon atoms confirms removal of impurities and metal nano particle used during synthesis of MWCNTs.

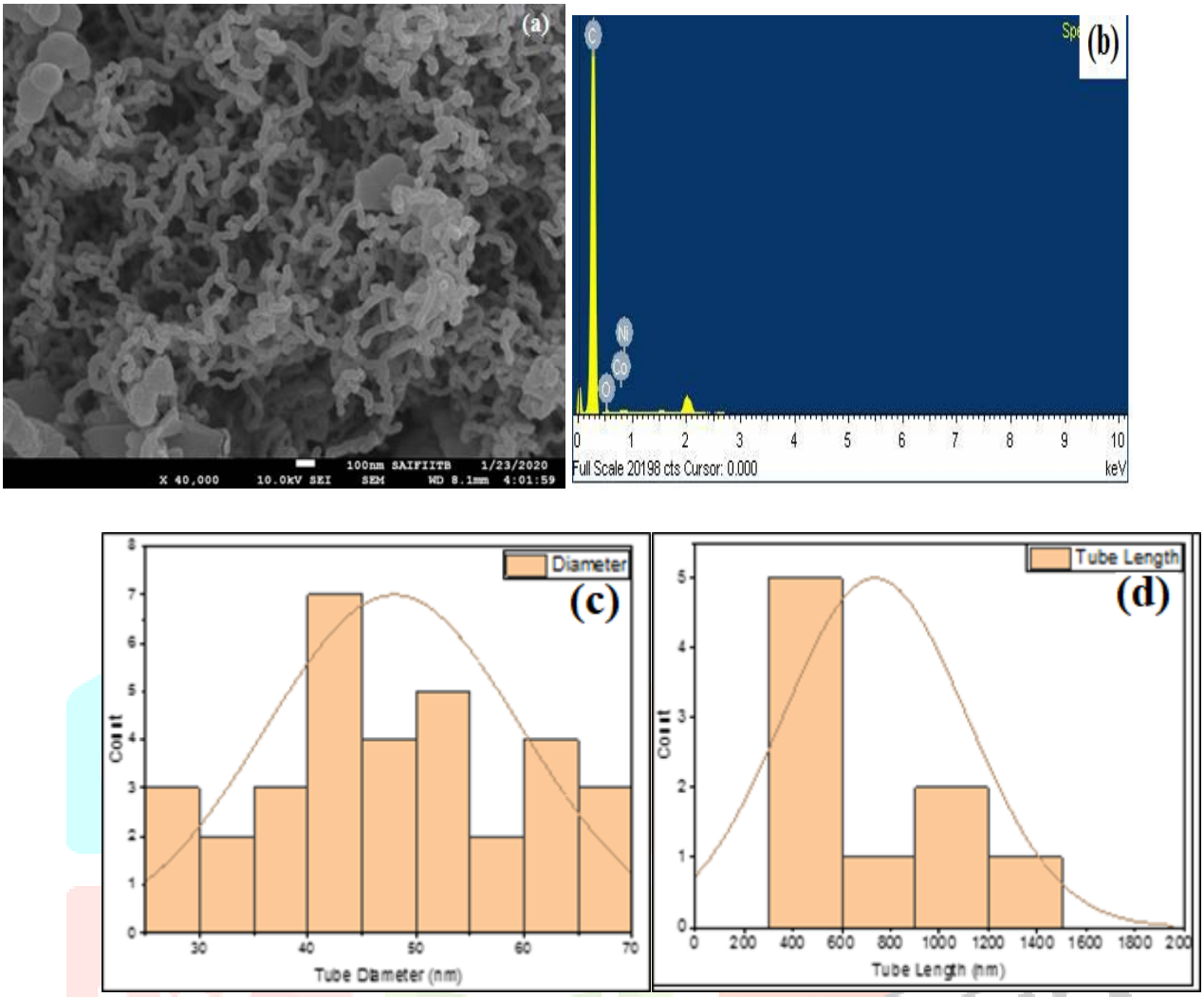

Figure 7. a) SEM image (b) EDX spectra (c) Diameters Histogram and (d) Length histogram of MWCNTs synthesized from Olive oil

TEM Analysis:

Figure 8. (a), show the TEM image of purified MWCNTs synthesized from Olive oil confirmed the tube-like structure of CNTs are multiwall and that the diameter of the synthesized MWCNTs was about 50-70 nm an open tube was seen at the tip. Figure 8 (b), shows the SAED image of purified MWCNTs. The SAED patterns of MWCNTs confirmed the graphitic nature of MWCNTs. The innermost ring which is due to the usual strongest reflection plane (002) of graphite. As shown in figure 8 (b), the plane (002) is indicated in the SAED pattern of obtained CNTs, which is consistent with the XRD peak at $2\theta = 26°$ plane. In this pattern, the bright spot and broad continuous rings could be attributed to small crystals. From these results, it could be suggested that the obtained MWCNTs were graphitic in nature and well degree of reorganization of crystallinity(Jagtap et al., 2013).

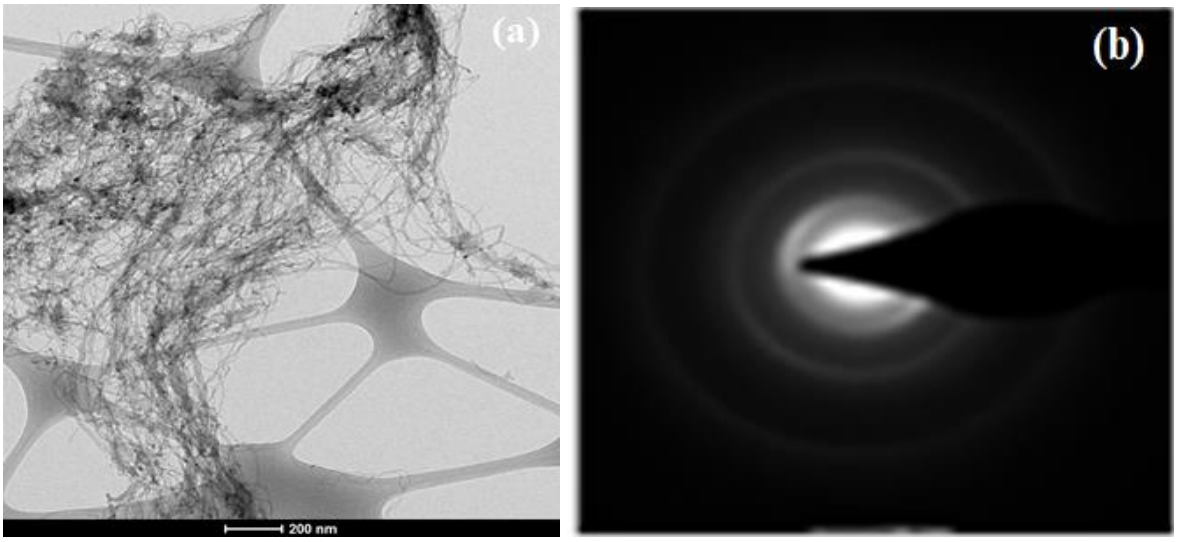

Figure 8. (a) TEM image and (b) SAED image of MWCNTs synthesized from olive oil

TGA Analysis:

Thermogravimetric (TGA) and Derivative thermogravimetric (DTG) analysis was carried out for investigating the thermal stability and purity of MWCNTs. Sample was heated under the dry Nitrogen flux from room temperature to 800^{o}C with heating rate 10^{o}C min^{-1}. As shown in Figure 9, decomposition processes and wet loss stared of MWCNTs start at 499^{o}C to about 750^{o}C that indicate MWCNTs was stable up to 750^{o}C.

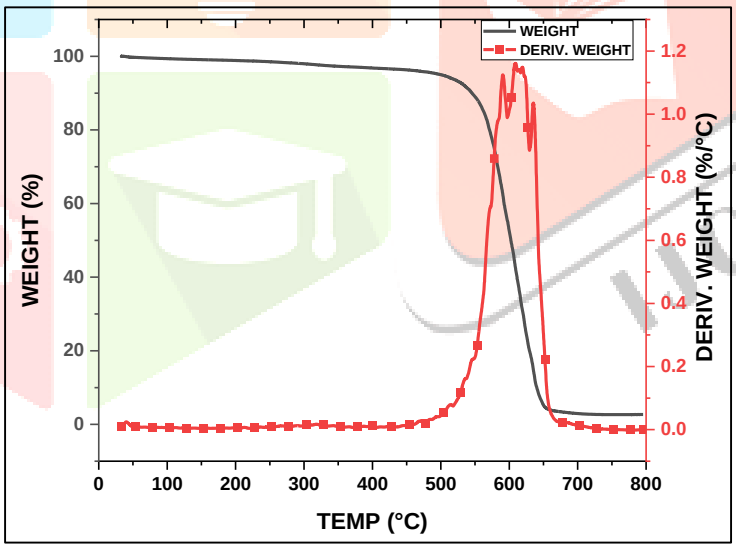

Figure 9. TGA Analysis of MWCNTs synthesized at optimal condition

There are apparently several combustion regions observable from the TGA graph which may be credited to the decomposition of carbonaceous materials along with the MWCNTs. The DTA profile show both exothermic and endothermic processes occurring simultaneously resulting in thermolysis of the solid. Exothermic peaks at 588,611 and 636^{o}C could be attributed as oxidation processes in the MWCNTs.

Surface Area Analysis:

The specific surface area of the MWCNTs is obtained by the N_2 adsorption using the BET method. The specific surface area of as grown MWCNTs was 96.78 m^2/g. After purification enhancement in surface area are seen which 193.49 m^2/g.

4.1 Electrical Conductivity:

Electrical conductivity of the MWCNTs was measured at room temperature in air using Four-Probe method. The conductivity obtained using this method are direct current (DC) conductivities, which can be calculated using measured resistance. Pellets of 2 mm thick and 10 mm diameters are used for measuring the electrical conductivity. Pellets are made by compressing MWCNTs powder by applying 100 MPa pressure using hydraulic press, which improve the contact between the carbon Materials. Pressed pellets are accessible for most solid-state materials, and are advantageous because their size is often larger than that of single crystals. Equation used for the determination of electrical connectivity are as follows:

The resistivity of the materials can be calculated by using the below equation:

$$\boldsymbol{\rho} = \frac{\rho_0}{\int(\frac{W}{S})} \text{----------2.1}$$

$$\rho_0 = \frac{V}{I} \; X \; 2\pi S \text{----------2.2}$$

and the conductivity can be determined using the equation:

$$\sigma = \frac{1}{\rho} \; S/cm \text{----------2.3}$$

The obtained electrical conductivity was 0.477 S/cm.

Conclusions:

In this study, we demonstrated Taguchi robust design method with L9 orthogonal array implemented to optimize experimental conditions for the synthesis of MWCNTs. Reaction annealing time is the most important parameter which affecting the yield and morphology of MWCNTs.

The best parameters for the synthesis of MWCNTs, when olive oil pass over Ni-Co NPs at 900 ^{o} C temperature and annealed for 120 min. in inert atmosphere, it gives 50 %. The synthesized MWCNTs having diameter in the range of 30-70 nm and length ranging from 400-1400 nm. The average diameter and length of MWCNTs are 48 nm and 736 nm, respectively. EDs confirmed the purity of 100 % MWCNTs. Electrical conductivity of the MWCNTs was measured at room temperature in air using Four-Probe method. The obtained electrical conductivity was 0.477 S/cm.

Future attention of this study for synthesized MWCNTs is in field of the energy applications and microwave absorption application. MWCNTs will also be used as light weight structural component with energy devices, batteries. This method will be providing light in weight and cost effective, ecologically friendly alternative methods.

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