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Optimization of LPG Diffusion Flame Synthesis of Carbon – nanotubed Structures using Statistical Design of Experiments (SDOE)

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Abstract— A statistical designed experimental approach was followed to investigate the various diffusion flame conditions for the synthesis of carbon nanotubed structures utilizing domestic Liquefied Petroleum Gas (IS – 4576) as the fuel carbon source. LPG flow rate, Oxygen flow rate, Height above burner (HAB) and Exposure time have been identified to generate different flame conditions based on varying one factor at a time (OFAT) approach. 2 – level 'full factorial' design model in "Minitab12" software has been used for design of experiments. The 16 samples of soot with different flame conditions were collected on the surface of stainless steel plate. The output parameters i.e. weight of soot and substrate surface temperature were analyzed by Analysis of variance (ANOVA) to ensure that the experimentation is following the physics of the diffusion flame. Transmission electron microscope (TEM) images showed the growth of well aligned single walled carbon nanotubed structures with high aspect ratio as 220 nm – 650 nm diameter and 534 nm – 1803 nm length. In the present study the parametric range for producing the single walled carbon – nanotubed structures through LPG diffusion flame were found and optimized.

Index Terms— Flame synthesis, carbon nanotubes, pyrolysis, DOE, LPG, Single walled carbon nanotubes, diffusion flame

1 Introduction

FLAME synthesis is emerging as a viable commercial mass production method for carbon nanotubes (CNTs). All the other known production methods such as Arc Discharge, Chemical Vapour Deposition (CVD) and Laser Ablation are energy intensive and lack in bulk synthesis of CNTs. The basic ingredients for all the processes are: a hydrocarbon fuel, a heat source and catalyst particles. Flame synthesis is a cost effective method since the flame serves as both the heating and the carbon source. The inner region is fuel rich and where soot formation dominates. The outer region is where oxidation dominates. The fuel-air mixing has a strong effect on the combustion efficiency, flame length, and the composition of the combustion products. Soot is the particulate formed during combustion of carbonaceous fuels under sub – stoichiometric conditions. In case of hydrocarbon fuels, soot is produced according to:

$$C_xH_y + zO_2 \rightarrow 2zCO + (y/2)\,H_2 + (x-2z)\,C \qquad (1)$$

and should occur when x becomes larger than 2z that C/O ratio exceeds unity but the onset of luminosity occurs when C/O is about 0.5 [1][2]. Thus soot volume fractions, mean particle diameters, and soot concentrations all depend critically upon both the radial and axial position within the flame [3]. If the region of large fuel concentration could be shifted to high temperatures then pyrolysis and soot or carbon nanotubes initiation chemistry can be accelerated [4]. Flames offer many variables for control that include fuel-air mixing, chemical environment and temperature. Parameters such as the nature of catalyst, growth times and gas flow rates affect the nature and yield of carbon nanotubes [5]. The fuel molecular constitution and the flame temperature govern soot and carbon nanotubes formation in laminar diffusion flames [6].

Formation of fibrous carbon in diffusion flames was first observed by Saito et al. [7], [8] while conducting soot characterization studies on methane air diffusion flames. The growth was observed beyond a certain height above the burner with associated color change from brown to black. Later, Yuan et al. [9], [10], [11] completed detailed characterization studies using methane – air diffusion flame. Yuan et al. also utilized ethylene – air co – flow and N_2 diluted ethylene – air diffusion flames to produce carbon nanotubes [12]. In the experiments done with methane and ethylene by Yuan and co-workers tried to study growth mechanism for the carbon nanotubes inside the flame using Scanning electron microscope (SEM) images and suggested that most nanotubes have a particle attached at the base near the substrate. Since the particles were not seeded in the flame and the images indicated their presence it is apparent that the particles are lifted from the surface. The base location of most of the particles supports the base growth model for the CNTs. Nevertheless, some particles were found at the tip of the nanotube indicating catalyst surface breakup. The effect of N_2 addition to the flame on the growth of nanotubes was assessed. Soot instead of CNT formation occurred when a bare stainless steel mesh was used as the substrate. However, when pre-oxidized substrate was used CNT growth was visible

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indicating the criticality of formation of metal oxides for CNT growth.

This phenomenon has been reported by Vander Waal et al. in their reports on premixed flame synthesis especially with an uncoated stainless steel mesh [13], [14], [15], [16], [17]. Vander Wal et al. utilizing a diffusion flame system produced SWCNTs at atmospheric pressure. Soot was formed when only Ni-Cr wire was held in the flame without any support mesh. This indicated that the stainless steel mesh may be essential for the formation of CNTs. Soot was found to grow over a broad range of conditions in the flame whereas CNTs grow in a narrower region in the presence of a catalyst. The CNTs were collected on Ni-Cr wire whereas brown deposits were formed on the stainless steel mesh later identified as iron oxide. CNT formation was observed even at low catalyst concentration indicating that soot formation and CNT growth may be competing phenomenon in the flame. Optimum region for CNT growth was found to be in the region of minimum oxygen concentration. Thus, it was suggested that oxygen might play an important role in the formation of metal catalysts. However, high concentrations of oxygen might lead to oxidation of the CNT precursors and incipient CNTs. The zone of temperature was also determined to be a critical parameter for the synthesis of CNTs.

Counter flow diffusion flames are been increasingly used for synthesis of MWNTs due to their 1-D geometry and convenience in positioning the catalyst substrate in the flame [18], [19], [20], [21], [22], [23], [24], [25]. Merchan-Merchan et al. [22] recorded the formation of CNTs in a methane oxygen counter diffusion flame without any catalysts. They employed an atmospheric, opposed flow burner with N_2 co-flow in which the oxidizer was enhanced to 50% oxygen or greater. High resolution SEM and TEM images revealed soot like structure with presence of carbon nano-particles and nanotubes however, no catalyst particles were found embedded in the soot like structure. The tube diameter and length were approximately 20 and 320 nm, respectively. Presence of nano-particles and nanotubes inside soot like structure pointed towards a similar mechanism responsible for formation of all three structural forms. Currently, oxy-flames are being pursued for CNT synthesis [18], [23] due to the high temperature and radical concentration obtained at the flame location.

Bharj et al. [26] used domestic LPG as fuel and the flame was generated using a gas welding torch to produce aligned SWCNTs. A stainless steel plate of thickness 2.5 mm has been used as substrate to collect the soot produced after the combustion of LPG. CNTs of length 325 nm to 616 nm corresponding to 2 mm diameter of nozzle at 140 mm stand off distance, substrate surface temperature of 625 K and flow rate of 1.8 LPM were obtained.

In diffusion flames CNTs and related nanostructures can be synthesized within the sooting zones present in the flame. Flames produced in various configurations and under different sets of conditions have been investigated for the synthesis of carbon nanotubes and related nanostructures.

The traditional experimental strategy for optimization of any synthesis process is the OFAT (One Factor At a Time) approach. In this approach, one of the parameter remains constant and all others are variables turn by turn. The response of the system is studied as a function of the changing variable. The major shortcoming of this approach is the amount of necessary experiments grows very fast with the number of variables and levels of variation of each variable, thus, the complete optimization of real systems is rather unfeasible. The reason for this is that OFAT assumes that the effects of variables are completely independent, whereas the response of a real system to change in any single parameter appears often as the gross effect of several parameter alterations. Statistical design of experiments (DOE) is the science of obtaining the largest possible amount of information about a system with smallest number of experiments [27].

In the present study, we proved that the DOE approach can be utilized successfully for the rapid optimization of flame synthesis of carbon nanotube growth. This study has been envisaged for the investigation of soot generated through domestic hydrocarbon fuel such as LPG (IS – 4576) by a simple continuous diffusion flame on a co – flow burner under normal environmental conditions in order to better understanding about the parameters controlling the formation of carbon nanotubes. Analysis of variance (ANOVA) is used to analyze the interaction of various parameters to the response (Weight of soot and Substrate surface temperature) by making the interaction plots in Minitab 12. The formation of CNTs was confirmed by Transmission Electron Microscopy (TEM). The goal of the optimization is to achieve the best parametric conditions of flame synthesis for growth of CNTs for specific carbon deposit quantity.

2 EXPERIMENTAL

2.1. Experimental setup

The experimental tests were carried out in a combustion test rig to study the correlation between flame conditions and the yield of CNTs. Fig. 1. shows the schematic diagram of experimental setup. The experimental setup consisted of the co-flow burner, substrate plate, traverse mechanism, acrylic sheet, gas cylinders (oxygen & LPG), rotameters, valves, hose pipes, thermocouple and thermocouple readout. The combustion takes place inside a transparent enclosed frame so that the flame is protected from the effect of surroundings and to allow the optical vision to the flame. The upper part of the frame is open to allow the exhaust gases to escape from inside the frame. The burner consists of simple tube – in – tube configuration. Two concentric tubes are arranged of diameters 10 mm and 50 mm respectively of length 200 mm. The traverse mechanism for the movement of the substrate is placed at the upper side of the frame. The base plate of traverse mechanism slides over the channels so as to move the assembly left & right. The substrate is a stainless steel plate of size 203 × 203 mm Grade 316 L. Gas supply system is designed to store and

supply the fuel and oxidizer to the combustion chamber. LPG (IS – 4576) is used as fuel and pure oxygen (99.9% pure) is used as oxidizer. LPG cylinder is having maximum storage capacity 44 liters at 2 MPa pressure and oxygen cylinder can store 20 kg oxygen by weight at 700 MPa pressure. Two rotameters are used to measure the volume flow rate of the LPG as well as Oxygen both having range 0 – 25 liters per minute (lpm). A thermocouple wire (Al – Cr, K – Type) is attached on the substrate plate to sense its surface temperature on which the soot has to be deposited. The numerical value of temperature is displayed on a calibrated K – Type thermocouple readout between the range of 0 – 1200 °C with maximum linearity error ±1.

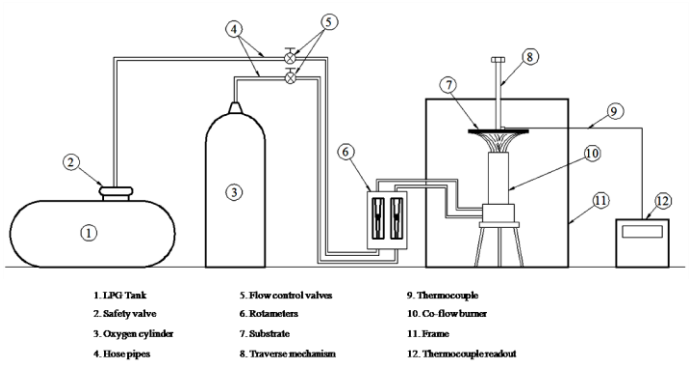
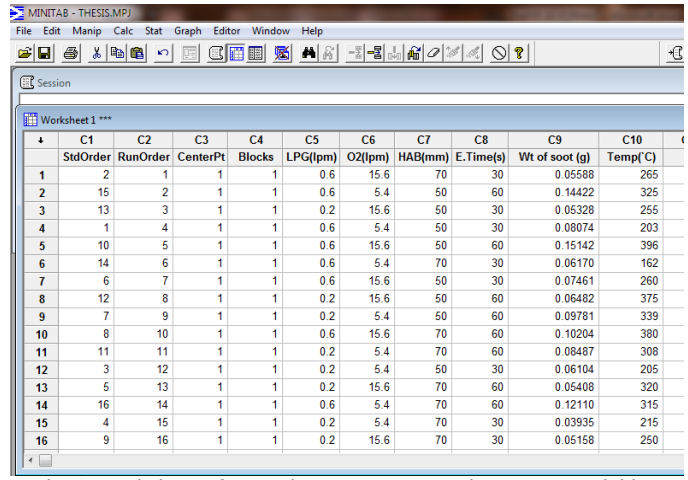


Fig. 1. Schematic of experimental setup

2.2. Design of experiments (DOE)

When analyzing a process, experiments are often used to evaluate in which process inputs have a significant impact on the process output, and what the target level of those inputs should be to achieve a desired result (output). Designed Experiments are powerful tools to achieve cost savings by minimizing process variation and reducing rework. In the present study, there are four parameters of the experimentation i.e. LPG flow rate, Oxygen flow rate, Height above burner (HAB) and Exposure time. There can be many numbers off levels of variation of each parameter for the experimentation. For example: if we take 3 levels of each parameter the maximum possible number of experiments to be performed are 81. In actual practice each and every possible combination of different levels of these parameters is difficult to investigate and evaluate. 'Minitab 12' offers four types of designed experiments: factorial, response surface, mixture, and Taguchi (robust). The requirement of the present experimentation is to optimize the variation each and every parameter for the growth of CNTs with the minimum number of experiments without repetition. Efficient optimization requires the early identification of key process parameters. This can be achieved by assuming that all parameters generate a predominantly linear response which is measured by setting each parameter to a ''low'' and a ''high'' value. Such a ''full factorial'' design is usually unfeasible for real-life systems since 2^k runs are required for k parameters. So, 2 – level 'full factorial' design model is used with 4 factors. Minitab automatically generates the run order of all possible combinations. The minimum and maximum level of numerical values of each parameter is decided on basis of initial trials performed before the actual experimentation. In the initial trials the minimum values are decided at which the soot start depositing on the substrate and the maximum value is decided when the soot particles tends to burn out. Minitab automatically generates the run order of all possible combinations. Total 16 combinations flame parameters generated by the Minitab. The experiments can now be performed according to the generated run orders and obtain the desired Response (output).



MINITAB - THESIS.MPJ

Worksheet 1 ***

	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
	StdOrder	RunOrder	CenterPt	Blocks	LPG(lpm)	O2(lpm)	HAB(mm)	E.Time(s)	Wt of soot (g)	Temp(°C)
1	2	1	1	1	0.6	15.6	70	30	0.05588	265
2	15	2	1	1	0.6	5.4	50	60	0.14422	325
3	13	3	1	1	0.2	15.6	50	30	0.05328	255
4	1	4	1	1	0.6	5.4	50	30	0.08074	203
5	10	5	1	1	0.6	15.6	50	60	0.15142	396
6	14	6	1	1	0.6	5.4	70	30	0.06170	162
7	6	7	1	1	0.6	15.6	50	30	0.07461	260
8	12	8	1	1	0.2	15.6	50	60	0.06482	375
9	7	9	1	1	0.2	5.4	50	60	0.09781	339
10	8	10	1	1	0.6	15.6	70	60	0.10204	380
11	11	11	1	1	0.2	5.4	70	60	0.08487	308
12	3	12	1	1	0.2	5.4	50	30	0.06104	205
13	5	13	1	1	0.2	15.6	70	60	0.05408	320
14	16	14	1	1	0.6	5.4	70	60	0.12110	315
15	4	15	1	1	0.2	5.4	70	30	0.03935	215
16	9	16	1	1	0.2	15.6	70	30	0.05158	250

Fig. 2. Worksheet of operating parameters and response variables

2.3. Sample preparation

Experiments were being performed as per the run orders, generated by Minitab, taking the respective parametric combinations of flame parameters. Normal Diffusion Flame (NDF) was created by the ignition of domestic LPG on the burner. Flame was enriched with pure oxygen to stabilize the flame as well as to change the flame conditions. Soot gets deposited on the substrate at various conditions of the flame.

2.4. Sample purification

Cross – flow micro – filtration technique is used to remove the impurities from the carbon material collected at the substrate. Then the samples were prepared by dispersion of the carbon material in ethanol and put under ultrasonication for 30 minutes.

2.5. Sample characterization

The drops of dispersed material were placed on copper carbon coated grid after negative staining and dried for TEM analysis. TEM micrographs were recorded on high resolution "Philips CM-10" electron microscope instrument for the analysis of the deposited carbon material under "Sophisticated Analytical Instrumentation Facility (SAIF)" at Department of Anatomy, All India Institute of Medical Sciences (AIIMS), New Delhi.

3 Results and Discussion

The numerical values of each response, obtained during the experimentation, were again put in the corresponding row in

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Minitab worksheet for the further analysis which is shown in Fig. 2. Analysis of variance (ANOVA) is used to analyze the interaction of various parameters to the response (i.e. Weight of soot and Substrate surface temperature) by making the interaction plots in Minitab. Minitab automatically generates the interaction plots for both the responses by performing statistical analysis.

Fig. 3. and Fig. 4. shows the interaction plots for Weight of Soot and Substrate Surface Temperature respectively. Since we were interested in tuning the system towards high SWCNT yield, it requires the screening of unimportant variables from interaction point of view. From the interaction plots, it was observed that the oxygen flow rate and exposure time can be take as primary factors and LPG flow rate and HAB as secondary factors. Interaction plots shows that oxygen flow rate and exposure time are the two most important factors for variation of substrate surface temperature whereas LPG flow rate and HAB mainly determines the quantity of soot deposited and sooting zone respectively.

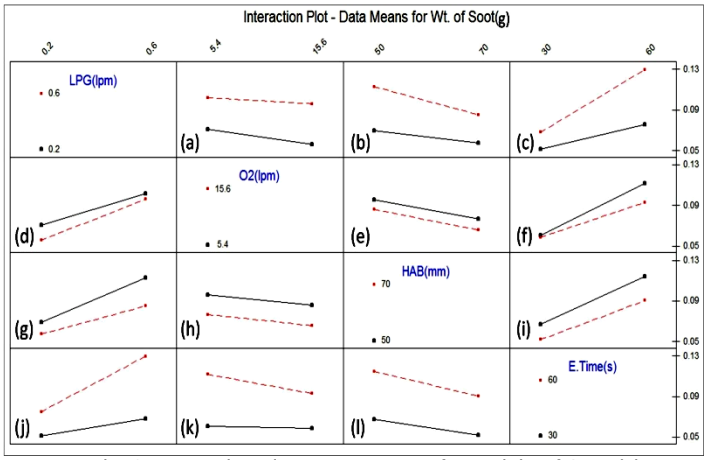

Fig. 3. Interaction plot – Data means for Weight of Soot (g).

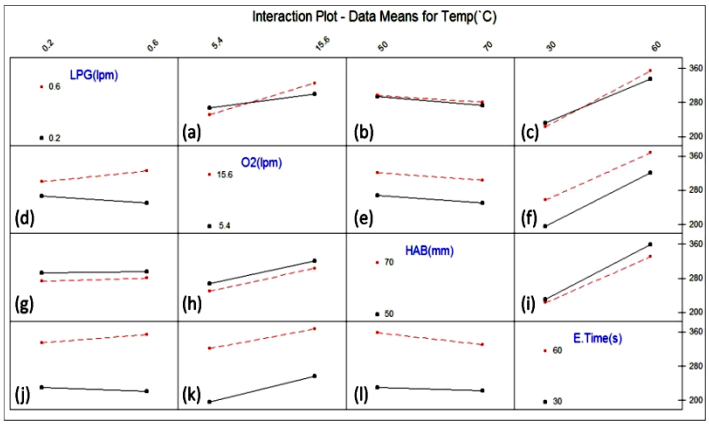

Figure 4. Interaction plot – Data means for Substrate Surface Temperature

Fig. 5. shows TEM micrographs of all the 16 samples collected after experimentation. The observations made from these images are discussed as follows:

3.1. Effect of flow dynamics

It was observed that the flow rates of LPG and oxygen significantly influence the formation and characteristics of carbon nanostructures synthesized in different flame environments.

When LPG flow rate of 0.2 lpm is made with oxygen flow rate of 5.4 lpm, a well mannered growth of nanostructures was observed. The TEM images of the samples which include these particular proportions of flow rates of LPG and oxygen appeared with more clarity. The clarity of images indicates to the purity of the nanostructures. On the other hand when the same LPG flow rate was combined with the oxygen flow rate of 15.6 lpm very thin nanostructures were observed. So it can be assumed from the physics of flame that when the oxygen flow rate was more, the gas phase temperature in the flame is more due to which the size of the nanoparticles become small before the growth takes place and the grown nanostructes also very small in size as compared to others.

Similarly when the LPG flow rate of 0.6 lpm is made oxygen flow rate of 5.4 lpm the concentration of carbon is more as compared to LPG flow rate 0.2 lpm with same oxygen flow rate. The growth of nanostructures was observed in some samples and some of the samples come out with soot particles or under grown carbon particles. TEM images of samples of these particular proportions of LPG and oxygen flow rate indicates thick and dense nanostructures. Therefore it can be assumed that the samples may either contain burnt particles which could have been separated during the filtration or thick soot particles. On the other hand when the same LPG flow rate is made with oxygen flow rate 15.6 lpm, the relative flow rate almost becomes same as with LPG flow rate 0.2 lpm and oxygen flow rate 5.4 lpm. TEM images of these samples, indicates that the nanostructures are under grown.

3.2. Effect of sampling technique

Effective sampling was decided during the initial trials before the actual experimentation. According to visual observation the maximum visible flame length was approximately 75 to 90 mm. Substrate has to be placed within the luminous zone which starts somewhere above the burner and below the tip of the flame. The minimum exposure time was decided when the layers of soot starts depositing and the maximum exposure time was decided at which the soot deposited starts burning.

It was noticed that when the substrate was placed above the flame for 30 seconds, either there was no growth of nanostructures or there are under grown nanostructures and when the substrate was placed above the flame for 60 seconds, most of the samples have shown sufficient growth of nanostructures. In case of the samples taken at 70 mm HAB, most of samples showing growth of nanostructures are either not aligned or their growth is non uniform. It was also noticed that the samples taken at HAB = 70 mm and for 30 seconds exposure time has not shown the growth of the nanostructures. The half of the samples taken at HAB = 70 mm and for exposure time 60 seconds have shown under grown carbon particles and half of the samples shown some growth

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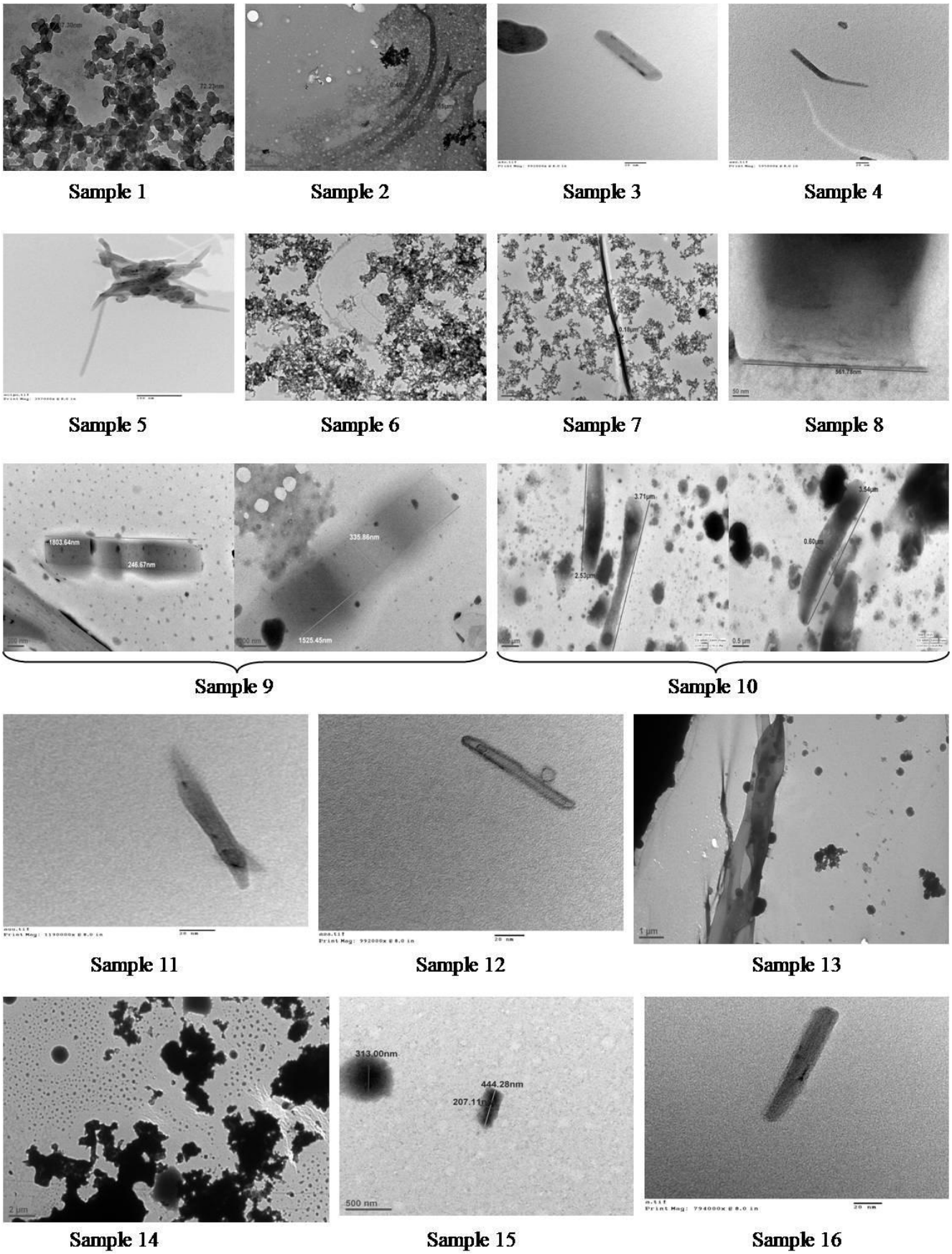

Figure 6. TEM images carbon nanostructures

but they are more dense and distorted or not aligned. The nanostructures of all the samples taken at HAB = 50 mm for 30 seconds exposure time are under grown but with 60 seconds exposure time, the sufficient growth of nanostructures was noticed.

3.3. Effect of substrate temperature

The experimental results showed that the substrate temperature also plays an important role in growth of CNTs. During the flame synthesis, the substrate temperature cannot be kept constant. Therefore the final temperature is recorded at the end of the set exposure time. The growth of CNTs have been observed when substrate surface temperature reached above 300 °C.

The effect of various experimental parameters on growth of CNTs is summarized in Table 1.

Table 2. Optimized parametric matrix for carbon nanostructures

Parameters	Parametric Range		Characterization of CNT structures
	LPG flow rate	Oxygen Flow rate	
Flow dynamics (lpm)	0.2	5.4	Well aligned large sized CNTs, diameter 220 – 650 nm
		15.6	Well aligned small sized CNTs, diameter 2 – 5 nm
	0.6	5.4	Dense chains of carbon nanoparticles
		15.6	Thick cylindrical carbon nanostructures
HAB (mm)	50		Well aligned CNTs
	70		Irregular and curved CNTs
Exposure time (seconds)	30		Initiation of CNT formation
	60		Well aligned CNTs, diameter 220 – 650 nm
Substrate Temperature (°C)	162- 215		No growth
	250 – 285		Nanoparticles, diameter 20 – 73 nm
	308 – 320		Irregular and curved CNTs
	325 – 396		Well aligned CNTs, diameter 220 – 650 nm

4 CONCLUSIONS

We reported the successful application of statistical design approach of experiment for optimization of flame synthesis of carbon structures. This experimental study represents the effects of various flame parameters and resulting substrate surface temperature on the formation of carbon nanotubes and nanostructures. TEM micrographs confirm the presence of CNTs in the collected samples.

- It was found that there is a specific requirement of LPG and oxygen concentration for the growth of CNTs. The growth of CNTs were observed with two combinations of LPG;Oxygen flow rates i.e. 0.2 lpm;5.4 lpm and 0.6 lpm;15.6 lpm respectively.
- The samples with the combination of LPG;Oxygen flow rate 0.6 lpm;5.4 lpm were showing thick and dense carbon particles. This is due to high carbon concentration during the combustion. Whereas the samples with combination of LPG;Oxygen flow rate 0.2 lpm;15.6 lpm were showing very thin carbon particles or small sized CNTs (~2nm diameter) were observed. Therefore the size of nanostructures (in diameter) is strongly influenced by LPG;Oxygen concentration during the combustion. Therefore, more fuel concentration during the combustion gives dense and thick carbon particles whereas more oxygen concentration gives small size carbon particles.
- The substrate surface temperature is also the important parameter which influences the growth of CNTs. It was observed that there were well grown and aligned CNTs when the substrate surface temperature is within the range of 325 – 396 °C.

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