Synthesis and Characterization of Carbon Nanotubes on Fe/Al_2O_3 Composite Catalyst by Chemical Vapour Deposition Method

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***Abstract*—This study reports the preparation and characterization of mono-metallic iron (Fe) catalyst on aluminum oxide (Al_2O_3) support for the synthesis of carbon nanotubes (CNTs). The Fe catalyst supported on Al_2O_3 was prepared using wet impregnation method and the process was optimized using factorial design of experiment approach. The influence of process variables (mass of support, stirring speed, drying time and drying temperature) were investigated. The catalyst sample with the highest yield (91.20%) after calcination obtained at drying time of 7 hours, mass of support of 10.50 g, stirring speed of 700 rpm and drying temperature of 110^{o}C was selected for subsequent CNTs synthesis, the sample was characterized using XRD, FTIR, SEM/EDS, BET and DLS techniques in order to determine the catalyst crystallinity, functional group(s), morphology, surface area/elemental composition and particle size respectively. The results obtained revealed that the catalyst has a specific surface area of 302.98 m^2/g. The morphology study of the catalyst with SEM indicated that the metal particles are evenly dispersed on the support with the particle size in the ranges of 100-1000 nm as revealed by the DLS analysis. FTIR spectrum indicated that the catalyst developed has two major two functional groups which are nitrate and hydroxyl group. The catalyst developed was utilized for the synthesis of CNTs by chemical vapour deposition method (CVD) with acetylene as the carbon source and argon as the carrier gas. The influences of production time and temperature on the yield of the CNTs were investigated. The CNTs synthesized was characterized using XRD, FTIR, SEM/EDS, TGA, BET and DLS techniques. The results obtained indicated that CNTs highest yield of 98.30% was obtained at production temperature of 750^{o}C. A nano-particle size within the range of 60.1 nm to 120 nm with highest abundance of 50 % was found to be presents in the CNTs with a surface area of 867.81 m^2g. The results also showed that the CNTs produced are thermally stable and TEM results indicated that they are multiwall carbon nanotubes. It can be inferred from various analyses conducted that the monometallic (Fe) catalyst on Al_2O_3 is a suitable catalyst for the production of good quality multiwall carbon nanotubes in a CVD reactor.**

***Keywords*—catalyst, CNTs, characterization, CVD, optimization.**

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I. Introduction

Nanotechnology is the science and technology of very small objects and phenomena on the scale of few to about 100 nm involving individual atoms, molecules, single electron, single photon [1-4]. Nanotechnology includes both the engineering of small scale as well as understanding of basic physical and chemical behaviour and phenomena of these objects. Nanotechnology is therefore, defined as a science of techniques used to modify and control matter at a very small scale [1]. It encompasses the technique and science utilized to operate atomically precise material and devices such as nanotubes photonic crystal quantum dots, lattice – strain device, nano - electronic mechanical structure [5, 6]. In chemical engineering, the technology pose the challenges of how to control molecules to form materials and how to apply this control to area such as assembly of surfaces, building structures atom, moving group of atoms and eventually connecting them. Hence, nanotechnology is important firstly because it has the potential to make optimal use of material and secondly because matter when modified at the nano scale can have extraordinary and useful properties which have never been observe before [7]. Using these properties will have the potential to create materials with benefit to health, welfare and prosperity of mankind. This is the reason why the science and technology of nanotechnology is importance. The tendency of nanotechnology to produce precise manipulation and control nanomaterials with unique properties make it to be influential technology that requires detailed understanding of physical processes across a range of discipline [8]. Therefore, the goal of nanotechnology is to produce new material, device and system tailored to meet the needs of growing range of commercial, scientific, engineering and biomedical applications opening new markets and giving dramatic benefits in products

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performance [9, 10]. Various types of nano particles such as carbon nanotubes, carbon nanoballs/ microsphere, carbon nanofibres, gold nanoparticle, silver nanoparticles and other metallic nanoparticle have been reported in literature. This present study will only focus on the carbon nanotubes. Different methods of production of carbon nanotubes such as laser ablation, arc discharge, flame synthesis, high pressure carbon monoxide, electrolysis and catalytic vapour deposition have been reported in literature [11]. Each of these methods has its drawn back, for instance it has been reported that arc discharge method produces large quantity of nanotubes but there is the problem of lack of control of alignment of carbon nanotubes produced by this method. Literature also revealed that laser ablation technique produces high yield of CNTs with low impurities however, the CNTs produced by this method is of non-uniform diameters which affects its applications in various field [12]. Though, catalytic vapour deposition method was identified as the best route of producing large scale quantities of CNTs with high yield due to the comparatively low growth temperature, however the nature of the catalyst also impede the CNTs produced by this method [13]. Hence there is the need to produce high qualities catalyst for the production of CNTs in CVD method for various applications.

Series of research have been conducted on the development of catalyst with transition metals on various supports for CNTs growth. The present emphasis on the catalyst development focuses on the development of bi-metallic catalyst and tri-metallic for CNTs growth. However, latest discovery indicates that CNTs produced on the bimetallic and trimetallic catalyst resulted into the low production of CNTs and in many cases large quantities of support are required which affect both the purity and cost of production [14]. Available literature on the synthesis of catalyst for CNTs production indicates that more is still required for proper understanding of interaction of factors such as the type of the metal catalyst, nature of support and others that influence the synthesis of catalyst for CNTs growth. Louis *et al.*, [15] reported that an enhanced dispersion of iron catalyst on Al_2O_3 is more effective compared to cobalt and nickel metal catalysts. Investigation have also shown that a better yield and quality carbon nanotubes can be produced from iron catalyst supported on aluminum oxide as the surface area and heating temperature of the supporting material has been studied to affect their growth [16]. With regards to study from various literature, it is evident that detailed optimization of the various process parameters namely mass of support, stirring speed, drying time and drying temperature have not been fully investigated for iron supported on aluminum oxide to the best of the author's knowledge, hence this research is aimed at optimizing the process of monometallic (Fe) catalyst on Al_2O_3 support for CNTs growth.

II. MATERIALS AND METHODS

All the chemicals used in this study are of analytical grade with percentage purity in the range of 98 – 99.99 % and the lists of chemicals used include aluminum oxide (Al_2O_3), iron (iii) nitrate nona hydrate ($Fe(NO_3)_3.9H_2O$). The distilled water was sourced from Centre for Genetic Engineering and Biotechnology. The gases are also of analytical grade with percentage purity of 99.9%. The approach used in this study involved preparation of Iron (Fe) catalyst on Al_2O_3 support by wet impregnation. The optimization of catalyst synthesis was carried out using a 2^4 factorial experimental design. The two (2) levels of the variables considered are as shown in Table1. The iron (Fe) catalyst on Al_2O_3 support was prepared by wet impregnation method, which is targeted mainly at dispersing the iron active components into the pores of the Al_2O_3 surface. The 2^4 factorial experimental designs was used to investigate the influence of mass of support, stirring speed, drying time and drying temperature using design expert software to generate the experimental matrix presented in Table 2. For the preparation of the catalyst, a calculated amount (5.656 g) of iron nitrate salt "$Fe(NO_3)_3.9H_2O$" was weighed and dissolved in 50 ml of distilled water. After which a required amount of Al_2O_3 support (9.50/ 10.50 g) was added under a constant stirring (300/700 rpm) for a period of 20 minutes. The resulting slurry was allowed to dry at room temperature after which it was oven dry at a known temperature of 110^{o}C for the low level and 120^{o}C for high level for a period of 5 and 7 hours respectively for low coded value and high coded value as presented in Table I. The product obtained was removed from the oven and cooled to ambient temperature, crushed and finally filtered through a 150 µm sieve. The fine powder obtained was then calcined at 400^{o}C for a period of 3 hours and the dried catalyst obtained was grounded to avoid agglomeration. The yield of catalyst was calculated using the relationship presented in equation (1).

$$\text{Yield } (\%) = \frac{\text{Mass of catalyst after calcination}}{\text{Mass after oven drying}} \times 100\ \% \quad (1)$$

TABLE I
VARIATION OF PARAMETERS OF THE 2^4 FACTORIAL DESIGNS

Level	Drying temperature (^{o}C)	Drying Time. (hrs)	Stirring Speed (rpm)	Mass of support (g)
Low	110	5.00	300	9.50
High	120	7.00	700	10.50

The sample that gave the highest yield was analyzed to determine the morphology, crystallinity, thermal stability and particle size using SEM, XRD, TGA and Nanosizer respectively. FTIR analysis was also used to check the nature of the bond present in the catalyst.

Carbon nanotubes were produced by the decomposition of acetylene over the monometallic (Fe) catalyst on Al_2O_3 support in a horizontal tubular furnace reactor known as catalytic vapour deposition reactor (CVD) at operating pressure of 1 atmosphere as shown in Fig.1. The CVD consists of a quartz tube (52 mm internal diameter, 4 mm thickness and 1010 mm length) with a heating capacity of about 1200^{o}C. Gas cylinders for the carbon source (acetylene) and the carrier gas Argon were connected to the inlet of the reactor where flow meters war placed to control the flow of gasses. A known amount (1.00 g) of the developed catalyst was dispersed in a quartz boat and was placed at the center of the quartz tube. The system was purged with argon at a flow rate of 30ml/minutes. Once the system attained the set temperature of 650 to 850^{o}C for the purpose of investigating the influence of temperature on the yield, the flow rate of argon was increased to 240 ml/min

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and the carbon source (acetylene) was open at regulated flow rate of 100 ml/min. The reaction was allowed to proceed for a period of 15-75 minutes with step increment of 15 minutes for the purpose of investigating the influence of time on the yield of carbon nanotubes. The carbon source was switched off immediately when the set time was attained and argon flow rate was reduced to 30 ml/min to allowed the reactor to cooled down to remove temperature. The quartz boat was then removed with the black soot content and weighed to determine the quantities of carbon nanotubes produced. The percentage yield of carbon nanotubes was then calculated using the relationship presented in equation (2) [17]. The product obtained was analyses to verify the nature of the yield, thermal stability, crystallinity and surface area.

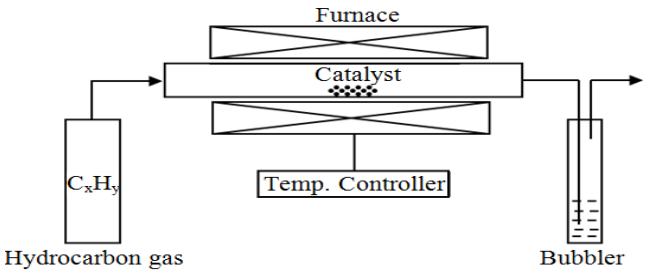

Fig. 1. Schematic of the CVD process

$$Yield\,(\%) = \frac{w2-w1}{w1} x\ 100 \quad (2)$$ where W_1 is the weight of the catalyst before reaction, W_2 is the weight of the catalyst + carbon deposit after synthesis.

III. RESULTS AND DISCUSSION

This study is focused on the investigation of process parameters (mass of support, drying temperature, drying time and stirring speed) on the yield of monometallic (Fe) catalyst on Al_2O_3 for possible application in carbon nanotubes growth. The results obtained (Table II) indicate that highest yield of 91.20% of catalyst was obtained at stirring time of 7 hours, mass of support of 1.50 g, stirring speed of 700 rpm and drying temperature of 110^{o}C.

TABLE II
VARIATION OF PARAMETERS OF THE 2^4 FACTORIAL DESIGNS

Mass of support (g)	Stirring speed (rpm)	Drying time (hrs)	Drying temperature (^{o}C)	Catalyst Yield (%)
9.5	300	5	110	83.40
10.5	300	5	110	85.40
9.5	700	5	110	78.00
10.5	700	5	110	85.40
9.5	300	7	110	89.00
10.5	300	7	110	90.00
9.5	700	7	110	84.40
10.5	700	7	110	91.20
9.5	300	5	120	81.40
10.5	300	5	120	78.40
9.5	700	5	120	81.80
10.5	700	5	120	60.00
9.5	300	7	120	78.40
10.5	300	7	120	76.00
9.5	700	7	120	83.40
10.5	700	7	120	76.20

The catalyst obtained at the best process condition was characterized to determine the basic properties such as the thermal stability, morphology, surface area, particle size and the nature of the bond presence.

Fig. 2 shows the thermal stability profile of the catalyst prepared. The results indicate three regimes of weight losses at 50^{o}C, 200-580^{o}C and 580–900^{o}C. The weight loss around 50^{o}C, could be attributed to the loss of absorbed moisture in the catalyst. The weight loss around 200-580^{o}C is as a results of evolution of Fe_2O_3 which is the product of interaction between iron salt and the support. The final weight loss around 580-900^{o}C could be attributed to the decomposition of the support (Al_2O_3) which indicates that the catalyst is thermally stable and will be suitable for the synthesis of carbon nanotubes at high temperature.

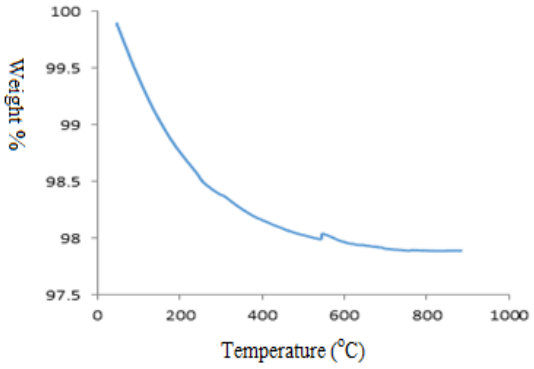

Fig. 2. TGA profile of the catalyst

Scanning electron microscope (SEM) was employed to study the morphology and elemental composition of the developed catalyst and the results obtained are presented in Fig. 3. From the Figure, it is seen that the catalyst is porous in nature with the metal particles well dispersed on the surface of the support. The EDS spectrum (Fig 3b) of the catalyst indicates that iron particles are present in the catalyst indicating that the wet impregnation method employed in this study is effective for deposition of iron metal on the surface of support. The EDS analysis shows that the sample contains high percentage of oxygen compared to other elements which is attributed to the decomposition of iron oxides (Fe_2O_3) during the calcination process [18]. Other elements present are the aluminum and carbon. The presence of carbon is as a result of utilization of carbon as a sample coating material for SEM analysis.

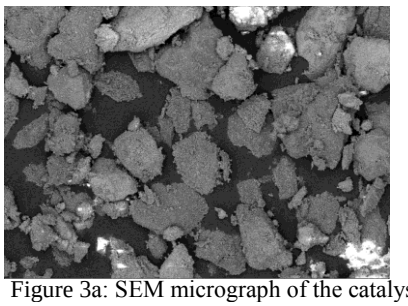

Figure 3a: SEM micrograph of the catalyst

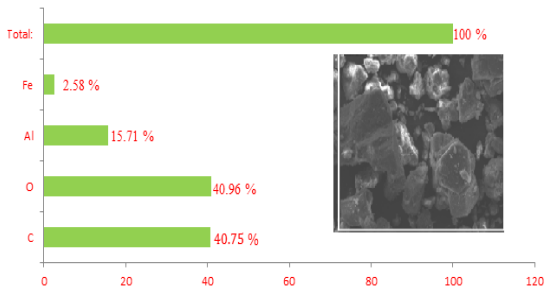

Fig. 3b. EDS analysis of the catalyst

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The specific surface area which is considered as an important property that determines the performance of homogenous catalyst was also determined using BET (Nova 4200e). The result indicates that the developed monometallic catalyst has a specific surface area of 309.98 m^2/g with pore volume of 0.1744 cc/g and pore size of 0.2954 Å. The particle size of the catalyst produced was determined using DLS technique. The results reveal that the catalyst has a particle distribution of 100-1000 nm, with average particle diameter of 721.4 nm, which also indicates that the catalyst prepared has the tendency of producing CNTs of nano diameter. The nature of the bond present in the catalyst was determined using FTIR. The FTIR spectrum is different from the near and far infrared and it is referred to as mid-infrared spectrum (400–4000 cm^{-1}) which is divided into four regions. It has been reported that the location of the region determines the nature of the functional group present.

Fig. 4 shows the FTIR spectrum of the catalyst. By convection, wavelengths in the ranges of 4000–2500 cm^{-1} are the fundamental vibrations region which are generally attributed to the O–H, C–H and N–H stretching. While the wavelength in the ranges of 3700–3600 cm^{-1} that produces a broad band due to the O–H stretching [20]. The results as presented in Fig 3 indicate that the two functional groups present in the prepared catalyst are the nitrate (NO_3^-) and hydroxyl (OH^-) corresponding to absorption band frequencies of 1290–1360 cm^{-1} and 2500–3600 cm^{-1} respectively which was compared favourably to the standard reported by Valentin [19].

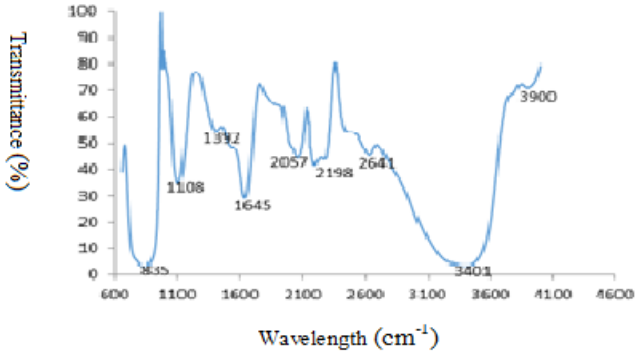

Fig. 4. FTIR spectrum of the catalyst

The monometallic (Fe) catalyst on Al_2O_3 prepared was used to synthesize CNTs by CVD method. The effects of time and temperature on the yield of the CNTs were investigated and the results obtained are presented in Figs 5 and 6 respectively. The influence of production time on the yield of CNTs was conducted by varying the time between 15 to 75 minutes at a constant temperature of 650°C. The results as presented in Fig. 5 indicate that the production yield increases with increase in time. However, the highest yield of CNTs was obtained at production time of 45 minutes, further increment in the production time beyond 45 minutes led to reduction in the yield of CNTs with lowest yield obtained at production time of 75 minutes. The results obtained on the effect of synthesis time on the yield of CNTs follow a specific trend reported by Afolabi et al. [13]. The group also reported in their work that shorter production time favours the production of CNTs with small diameter while longer growth duration favours the growth of short, thick and fibrous MWCNTs due to continuous deposition of the carbon on the catalyst thereby compressing the growth of CNTs.

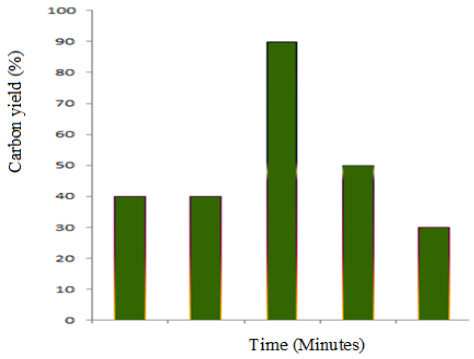

Fig. 5. Influence of time on the yield of CNTs

Growth temperature has been reported as an important parameter that affects the yield, purity, growth rate and morphology of CNTs produced by CVD method. It has been reported that variation in growth temperatures (500 – 1000°C) affect mainly the selectivity of the catalysts and high temperatures tend to produce nanotubes of poor quality. Thus, in order to improve production rate and quality CNTs, appropriate choice of temperature is vital to reduce the amorphous carbon and some other forms of impurities that may be formed during the reactions. In this study, the synthesis temperature was varied between 600°C to 850°C with step increment of 50°C and a period of 45 minutes been the best time was selected as the reaction time. At a temperature below 650°C production of CNTs was not observed, however at a reaction temperature of 650°C, a black solid powder was observed. The results in Fig. 6 also indicate that the yield of CNTs increases with increase in temperature between 650°C to 750°C. Further increase in temperature beyond 750°C resulted into reduction in the yield of CNTs. Hence, the growth of high carbon yield is favoured at an optimum temperature of 750°C which is closely related to the result of Alain *et al.* [20]. It has been reported that at high temperature, the catalyst particle will aggregate due to the decrease in the density distribution of the catalyst particles on the surface of the support thereby providing enough space for the diffusion of the carbon source to the metal particle which aid the production rate of CNTs. Literature also reported that at high production temperature there is the possibility of complete decomposition or dissolution of the carbon sources which also results in the increment production rate [8].

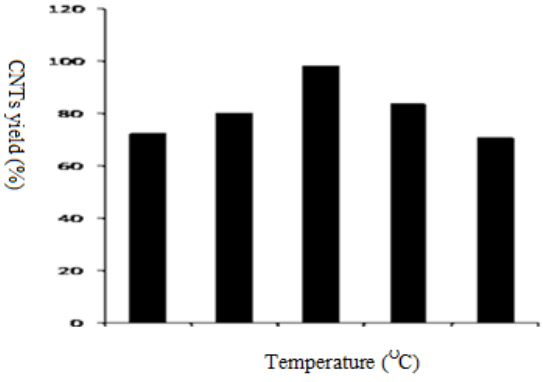

Fig. 6. Effect of temperature on the yield of CNTs

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However, increase in temperature beyond the limit of the sources will give the molecule of carbon source enough energy that will increase the velocity profile in the reactor thereby allowing the molecules of carbon source to escape without participating in the reaction. The black soot obtained from the CVD at best conditions of 45 minutes and 75°C was analyzed to determine the morphology, nature, thermal stability, crystallinity and specific surface area.

The images in Figs 7a & b represent the respective morphologies of CNT samples obtained at 650°C and 750°C at constant production time of 45 minutes. The image of the sample synthesized at 650°C contains some amorphous carbon which is absent from the sample obtained at 750°C. The SEM micrograph also shows that the sample obtained at 750°C are well arranged and not entangled as compared to the sample obtained at 650°C.

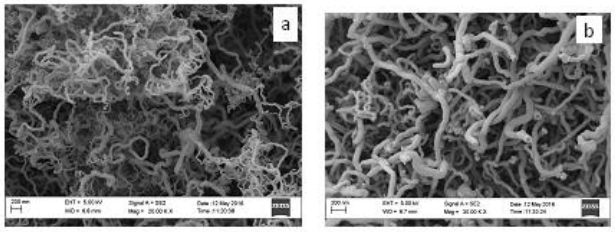

Fig. 7. SEM micrograph of the CNTs

Figs 8a & b are the TEM images of the CNTs at reaction temperature of 650°C and 750°C respectively. The results indicate that the products obtained are hollow in nature with inner diameter and length of several micrometers indicating that CNTs were synthesized. It can be also observed from the TEM images of the sample that the CNTs contain several walls, hence the CNTs synthesized are multiwall carbon nanotubes. The TEM image also reveals that CNTs synthesized at 650°C (Fig. 8b) contain large cluster of catalyst particles which is an agglomerate of metal that is present as impurities in the sample, which also collaborates the earlier observation in SEM micrograph. The TEM images also show that the sample obtained at 750°C are more aligned than the sample obtained at 650°C.

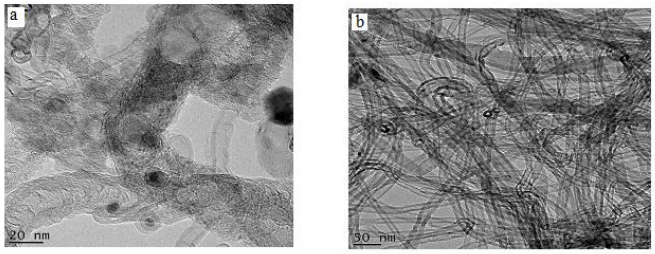

Fig. 8. TEM images of CNTs produced

The thermal stability of the CNTs synthesized was also studied and the TGA/DTA profile obtained (not shown) indicates that the CNTs are thermally stable. It has been reported that TGA analysis assists in examining the percentage weight loss and derivative weight percentage as a function of temperature [11]. The results indicate that the CNTs produced are thermally stable as the CNTs produced retained more than 86% of its original value at temperature of 1000°C indicating that the CNTs are highly graphitized. The pore size, pore volume and surface area of the CNTs produced was obtained using BET and the results indicate that the CNTs produced has a high surface area of approximately 868 m^2/g with pore size of 0.3128 Å and pore volume 0f 0.3486 cc/g which indicate that CNTs synthesized can be utilized as adsorbent.

The XRD was used to analyze the characteristic pattern of graphitized carbon and graphitic line observed around 26° for all samples which corresponds to an inter-planner spacing of about 0.343 nm which is usually attributed to graphite as reported by Afolabi *et al*, [11]. This pattern also indicates a high degree of crystallinity which suggests a low content of amorphous carbon and impurities from the catalyst which are usually metal particles. The XRD pattern was used to in conjunction with the Debye-Scherrer equation presented in equation (3) to determine the particle size distribution of the CNTs produced. The results obtained indicate that the theoretical particle size of the CNTs produced is in the ranges of 34 to 120 nm.

$$Size_{Scherrer} = \frac{K\times\lambda}{(FWHM)\times Cos(\theta)} \quad (3)$$

where FWHM is the full width at half maximum of a peak in the XRD pattern (in radians), K is shape factor called Scherrer constant with value between 0.2 and 2.0, λ is the X-ray wavelength of 0.154 nm used in the X-ray diffraction, and θ can be evaluated from 2θ values.

Aspect ratios are important factors that determine the applicability of CNTs in various areas. It is expected that CNTs with various lengths can be used in a variety of fields such as electronic, biological and composite materials. Hence, the aspect ratios of CNTs produced were obtained from the DLS results. Fig. 9 shows the correlation chart between the aspect ratio, length and diameter of the CNTs produced at 750°C using the relationship presented in equation (4).

$$D_h = \frac{L}{\ln(L/d)+0.32} \quad (4)$$

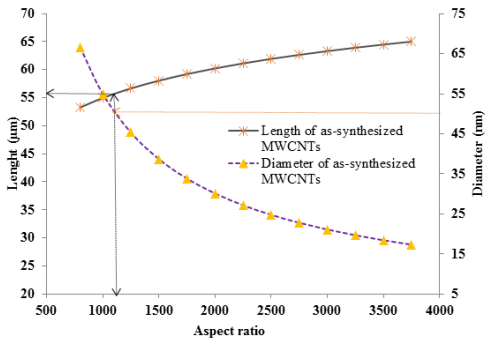

Fig. 9. DLS Correlation Chart of CNTs

The results indicate that the aspect ratio of CNTs produced was approximately 1129 with length of 55.84 µm, which is in agreement with the findings Rashid *et al*. [21] which states that the aspect ratio of MWCNTs synthesized by CVD process could range from 157 to 3750.

IV. Conclusions

This study was aimed at CNTs synthesis from the monometallic (Fe)/Al_2O_3 composite catalyst using CVD method. The analyses of the results obtained indicated that the operating parameters (mass of support, stirring speed, drying time and drying temperature) influenced the yield of catalyst. The highest yield of 91.20% of catalyst was

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obtained at drying time of 7 hours, mass of support of 10.50 g, stirring speed of 700 rpm and drying temperature of 110^{o}C. The results also revealed that the reaction time and temperature influenced the yield of CNTs by CVD method. The reaction time of 45 minutes and temperature of 750^{o}C were the best conditions for the synthesis of CNTs with monometallic (Fe)/Al_2O_3 composite catalyst in a CVD reactor. The analysis of the CNTs synthesized indicated that the CNTs were thermally stable, graphitized with high surface area which suggested their application in adsorption process. The TEM analysis also confirmed that the CNTs were MWCNTs with average aspect ratio of 1129 and average length of 55.84 µm. It can therefore be concluded from various analyses conducted that the monometallic (Fe) on Al_2O_3 support is a suitable catalyst for the synthesis of good quality CNTs in a CVD reactor.

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