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## KINETICS OF ACETYLENE DECOMPOSITION OVER REDUCED STRONTIUM HEXAFERRITES CATALYST FOR THE PRODUCTION OF CARBON NANOTUBES

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### ABSTRACT:

Carbon nanotubes were synthesized over Catalysis of the composition 40Strontium hexaferrites SHF ( $\text{SrFe}_{12}\text{O}_{19}$ ): 60 $\text{Al}_2\text{O}_3$  by the catalytic decomposition of acetylene for different reaction conditions. The kinetics of synthesis of CNTs were investigated through two types of experiments, the first was done at different temperatures. The optimum conditions for the higher yield % were found to be 600 °C which give 367 yield %. The second type of experiments was done at variable decomposition temperature and constant reduction temperature at 600 °C. The highest yield % was found at reduction and decomposition temperature 600 and 700 °C respectively (436.9%). The structure and morphology of the synthesized CNTs were characterized using high resolution transmission electron microscopy (HRTEM).

### KEYWORDS:

Carbon nanotubes, SHF, kinetics, and Acetylene decomposition.

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## INTRODUCTION

It is clear that one of the most active fields of research in the area of nanotechnology is synthesis, characterization and application of carbon nanotubes (CNTs) [1-3]. CNTs are graphitic sheets with internal channel and they have high electrical conductivity, extremely high mechanical strength and can be divided into two categories: single-wall carbon nanotubes (SWNTs) and multi-wall carbon nanotubes (MWNTs). CNTs have been observed in 1991 by Iijima [4]. CNTs have wide range of applications such as being a metal, a semiconductor, superconductor [5, 6], also their hydrogen storage capacity [7, 8], electronics [9], catalysis [10,11], and polymer science [12-14]. CNTs have been synthesized by numerous techniques, such as arc discharge [15, 16], laser ablation [16,], plasma-enhanced [17], and chemical vapor deposition (CVD) of hydrocarbon gases (methane, ethane, and acetylene) at rather high temperature over a catalytic material [16,18]. The yield %, and type of CNTs deposited depends on support type [19,20], mixture of bimetallic Fe/Co supported on CaCO<sub>3</sub> gives 358.7 % of very spongy voluminous carbon deposited while the same catalyst supported on MgO gives 229% of spongy carbon deposited. Also yield % depends on percentage of metal loading, at small Fe content (2.3 %) supported on silica small percentage of carbon deposited was formed, but at high Fe content (4.4%, 8%, and 28.5%) the amount of carbon deposited increase [21], and 5 and 10 wt % Co–Mo/MgO catalysts produced (6-27%) single-wall nanotubes respectively, whereas 20, 30 and 40 wt.% Co–Mo/MgO catalysts produced ( 567.8 % at 40wt %) multi-wall nanotubes [22]. The yield % increases almost linearly with increasing reaction temperature, Fe-Ni bimetallic catalyst supported on alumina, the carbon yield % increases from 74.9% at 600°C to 121.5% at 700°C, the higher reaction temperature result in the formation of CNTs with more stable structures [ 23]. The reaction time has great effect on yield %, at constant metal loading of bimetallic catalyst Fe-Ni supported on alumina yield % increases with increasing reaction time, but higher disorder carbon appears between 60-90 min. Smaller catalyst particles enabled the synthesis of highly dense, long and narrow-diameter CNTs. C<sub>2</sub>H<sub>2</sub> exhibits very high activity in producing metal carbide compared to CH<sub>4</sub> and CO [24]. It is notable that the generation of metal carbide probably hinders the formation of carbon nanoshells which poison the catalytic center [19]. Equilibrium is essential between the C<sub>2</sub>H<sub>2</sub> feed stock and the surface of catalyst particles during synthesise of CNTs, such equilibrium appeared at 30 sccm C<sub>2</sub>H<sub>2</sub> for Ni/Fe supported on Al<sub>2</sub>O<sub>3</sub> [25]. Fe containing catalyst efficiently promoted the formation of CNTs in CCVD process if compared by Ni and Co [26, 27], duo to with iron catalysts the interaction between formed carbon and metal particles is apparently less structure sensitive than with Ni or Co [28], this may be related to higher carbon concentration during the steady state growth for iron catalysts, its catalytic activity for the decomposition feedstock, the formation of meta-stable carbides, the carbon diffusion, and finally the formation of graphitic sheets. The aim of the work is to study the kinetic of the synthesis CNTs over freshly reduced strontium hexaferrites (SHF) supported on alumina at different temperature, reaction time, gas flow, heating rate, metal loading %, carrier gas, and pressure were identical in all experiments, while the reaction temperature was investigated in details.

## EXPERIMENTAL PROCEDURE

Catalyst of the composition 40SHF:60Al<sub>2</sub>O<sub>3</sub> is prepared by wet impregnation method. A high metal loading was chosen in order to obtain a longer catalytic activity. The catalysts were prepared as follows: aqueous solutions of SHF (which prepared by self flash combustion) with the required amount, was mixed with Al<sub>2</sub>O<sub>3</sub> powder and stirred for 1 hrs at 60 °C to remove dissolved oxygen and to achieve a homogeneous impregnation of catalyst in the support. The impregnate was then dried in an oven at 100 °C for 3 hrs, calcined at 400 °C for 4 hrs in a box muffle furnace. The produced supported catalysts were stored in a sealed vessel. Approximately 50 mg of a catalyst sample was introduced in to cylindrical alumina cell closed with one end, the cell with the catalyst placed in the central region of a longitudinal furnace. The catalyst was reduced at different temperature 500, 550, 600, and 650 °C at 1L/min in H<sub>2</sub> flow and CNTs were synthesized at the same reduction temperature by flowing 10C<sub>2</sub>H<sub>2</sub>:90H<sub>2</sub> to obtain the optimum temperature for high yield % of CNTs (600 °C). At constant reduction temperature (600 °C) the effect of growth temperature on the yield % was also examined by carrying out the reaction at temperature 500, 600, 700, and 800 °C. The synthesized CNTs were cooled in H<sub>2</sub> flow and the weight of deposited CNTs was detected using weight gain technique. The activity of catalyst was measured by yield % of carbon deposited which can be calculated from the following relation,

$$C\% = [W_3 - (W_1 - W_2) / (W_1 - W_2)] * 100 \quad (1)$$

Where W<sub>1</sub> is the initial weight of the catalyst, W<sub>2</sub> is the weight loss of catalyst at operating temperature, and W<sub>3</sub> is the weight of carbon deposited and catalyst.

The structure and morphology of the synthesized CNTs were characterized using high resolution transmission electron microscopy (HRTEM). The crystallite size was detected from X-ray diffraction using Scherer's equation using software Topaz 2, and confirmed by TEM.

## RESULTS AND DISCUSSION

### kinetics

There are many factors affecting the decomposition of hydrocarbon gases to carbon nanotubes (CNTs), the smaller catalyst particles enabled the synthesis of highly dense, long and narrow-diameter CNTs [24]. It was found that a homogeneous dispersion of the catalyst was an essential factor in achieving high growth density. The kinetics of synthesis of CNTs on reduced SHF supported on alumina were investigated through two types of experiments, the first was done at constant reaction time (30 min.) and rate of gas flow 10C<sub>2</sub>H<sub>2</sub>: 90H<sub>2</sub>. Samples were reduced at 500-650 °C and subjected to C<sub>2</sub>H<sub>2</sub> flow at each temperature to get the optimum conditions for the higher yield %. Temperatures have great effect on the yield percentage of carbon deposited, as show in Fig 1. The increase in reduction temperature from 500 to 600 °C increases reduction extent, so yield % increases from 171.3% at 500 °C to 367 % at 600 °C as shown in Fig 2. But by increasing reduction and decomposition

temperature from 600 to 650 °C the yield % decreases from 367 % at 600 °C to 329.4% at 650 °C, this can be attributed to grain growth for the resulted iron metal which increases its crystal size, as shown in Table 1. From the above data the optimum reduction temperature for the higher yield % is 600 °C, this can be attributed to increase in reduction temperature which led to increase in the formed iron metal % as shown in Fig 3, and Table 1, which has great tendency for the growth of CNTs if compared by Ni and Co [26,27]. This can be attributed to that, in iron catalyst the interaction between formed carbon and metal particles is apparently less structure sensitive than with Ni or Co. This may be related to higher carbon concentration during the steady state growth for iron catalysts, its catalytic activity for the decomposition feedstock, the formation of meta-stable carbides, the carbon diffusion, and the formation of graphitic sheets. All of these reasons enabled the synthesis of highly dense, long and narrow-diameter CNTs [24] as shown in Fig 4. The degree to which the metallic iron (resulting from reduction process) is dispersed on the support on reduction treatment is a function of the iron content [21]. By optimization of this parameter, it is expected that a maximum number of active points for acetylene decomposition can be achieved and a correlation of the structure of the generated CNTs with these metal particle sizes established.

The second type of experiments was done by increasing in decomposition temperature at constant reduction temperature at 600 °C that gave the highest yield %. This was done at reaction time of 30 min, rate gas flow of 10C<sub>2</sub>H<sub>2</sub>: 90 H<sub>2</sub>, and variable decomposition temperature 500, 600, 700, and 800 °C. The yield % increases by increasing decomposition temperature from 230.2% at 500 °C to 436.9 % at 700 °C, as show in Fig 5. In the above conditions we have constant phases content, so any increasing in yield percentage will be related to change in decomposition temperature. The lower decomposition temperature provides the optimum conditions for the formation of active metal sites with small crystal size, hence enable the formation of highly dense (Fig 6a) and narrow diameters CNTs (Fig. 6b). On the other hand at higher decomposition temperature (800 °C) yield % decreases to 180.7% because of the increasing in crystal size of iron metal as a result of grain growth at higher decomposition temperature. Not all the deposited carbon is CNTs, but it contains some biproducts like amorphous carbon, fiber, soot, and other carbon products.

Figures (7 and 8) show the Arrhenius plots of CNTs synthesis, these plots are straight lines with negative slopes, which give activation energy of 26.3 and 5.2 kJmol<sup>-1</sup> for the first and second experiments respectively. From the activation energy values the adsorption of acetylene on catalyst surface is a physisorption process, which involves the balancing of a weak attractive force. The second experiment has the lowest value of the activation energy which may be attributed to increasing in decomposition temperature.

## Surface Area Analysis

Table 2 shows the surface area measurements for the SHF supported on alumina with the molar ratio 40 (SrFe<sub>12</sub>O<sub>19</sub>): 60Al<sub>2</sub>O<sub>3</sub>. From this Table we can indicate that the catalyst has curie temperature around 500 °C, the catalyst has different behaviors below and above these temperature. At 500 °C the surface area was found to be 76.55 m<sup>2</sup>/g, which corresponding 171.3% carbon yield. The smaller yield % can be attributed to the lowest reduction and decomposition temperature, but above the curie temperature the surface area was found to be 64.39 m<sup>2</sup>g<sup>-1</sup> at 550 °C and 77.84 m<sup>2</sup>g<sup>-1</sup> at 600 °C which corresponding to the highest yield percentage 272.3 % and 367% respectively due to the high surface area which increases the dispersion of metal active sites on the support, by increasing the reduction and decomposition temperature to 650 °C the surface area decreases to 57.56 m<sup>2</sup>g<sup>-1</sup> due to an increase in the crystal size as a result of grains growth at higher reduction and decomposition temperature, so the carbon yield % decreases to 329.4 % and these are in a good agreement with the kinetic data.

From the adsorption isotherm, the catalyst is consider as type II isotherms which are the normal form of isotherm obtained with a nonporous or macroporous adsorbent and this was in a good agreement with the activation energy calculation. This type of isotherm represents unrestricted monolayer-multilayer adsorption, and it considers as type C of hysteresis loops as shown in Fig. 9 which is produced by wedge-shaped pores with open ends as confirmed by TEM in Fig. 10.

## Growth Mechanism

The way in which nanotubes are formed is not exactly known. The growth mechanism is still a subject of discussion, and more than one mechanism might be possible during the formation of CNTs. One of the mechanisms consists out of three steps. First a precursor to the formation of nanotubes and fullerenes, C<sub>2</sub>, is formed on the surface of the metal catalyst particle. From this metastable carbide particle, a rodlike carbon is formed rapidly. Secondly there is a slow graphitisation of its wall. This mechanism is based on in-situ TEM observations [29]. For supported metals, filaments can form either by 'extrusion (also known as base growth)' in which the nanotube grows upwards from the metal particles that remain attached to the substrate which not observed in our catalyst, or the particles detach and move at the head of the growing nanotube, labelled 'tip-growth' and this is in a good agreement with TEM image as shows in Figures 2 and 4. [30].

## Summary And Conclusions

1. Catalyst of the composition 40Strontium hexaferrites SHF (SrFe<sub>12</sub>O<sub>19</sub>): 60Al<sub>2</sub>O<sub>3</sub> was prepared by wet impregnation method. Carbon nanotubes were synthesized over the prepared catalysts by the catalytic decomposition of acetylene at different reaction conditions.

2. The kinetics of synthesis of CNTs were investigated through two types of experiments, the first was done at constant reaction time 30 min and rate gas flow of 10 C<sub>2</sub>H<sub>2</sub>: 90 H<sub>2</sub>, samples were reduced at 500-650 °C and subjected to C<sub>2</sub>H<sub>2</sub> flow at each temperature. The optimum conditions for the higher yield % were found to be 600 °C which give 367 %
3. The second type of experiments was done at variable decomposition temperature (500-800 °C) and constant reduction temperature (600 °C). This was done at the same experimental conditions. The highest yield % was found at reduction and decomposition temperature 600 and 700 °C respectively.
4. The activation energies for the first and second experiments were found to be 26.3 and 5.2 kJmol<sup>-1</sup> respectively, so the adsorption of acetylene on catalyst surface is physisorption process.
5. From the adsorption isotherm, the catalyst is considered as type II isotherms. This type of isotherm represents unrestricted monolayer multilayer adsorption, and it considers as type C of hysteresis loops which is produced by wedge-shaped pores with open ends.
6. The presence of catalytic nanoparticles at the tip of the produced CNTs suggests that the CNT production occurred via a tip-growth mechanism

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Table 1. Crystal size and percentage of phase content as shows from XRD analysis for completely reduced SHF pellets.

Reduction Temperature (°C)	phases content	phases content (%)	Crystal size (nm)
500	Sr <sub>4</sub> Fe <sub>6</sub> O <sub>13</sub> ,	50	62.1
	Fe <sub>21.4</sub> O <sub>32</sub> ,	50	32.2
	FeO,	50	23.7
	Fe (metal)	12.5	43.2
550	Fe (metal),	50	98.2
	FeO,	50	36.2
	Sr <sub>2</sub> Fe <sub>2</sub> O <sub>3</sub>	20.8	31.8
600	Fe (metal),	50	104
	Fe <sub>21.4</sub> O <sub>32</sub> ,	6.25	
	Fe <sub>2</sub> O <sub>3</sub> ,	10.4	
	FeO,	6.25	20
	Sr <sub>2</sub> Fe <sub>2</sub> O <sub>5</sub> ,	6.1	77.6
	SrO	4.17	

Table 2. The surface area measurements for the SHF supported on alumina with the molar ratio 40 (SrFe<sub>12</sub>O<sub>19</sub>): 60Al<sub>2</sub>O<sub>3</sub>.

Reduction temperature	500 °C	550 °C	600 °C	650 °C
Surface area (m <sup>2</sup> g <sup>-1</sup> )	76.55	64.39	77.84	57.56
Total pore volume (Ccg <sup>-1</sup> )	0.03788	0.03194	0.0368	0.02699
Average pore diameter (nm)	19.79	19.84	18.91	18.76
Micro pore volume (Ccg <sup>-1</sup> )	0.08814	0.07489	0.08045	0.0668
Adsorption energy kJmol <sup>-1</sup>	2.429	2.208	2.357	2.217

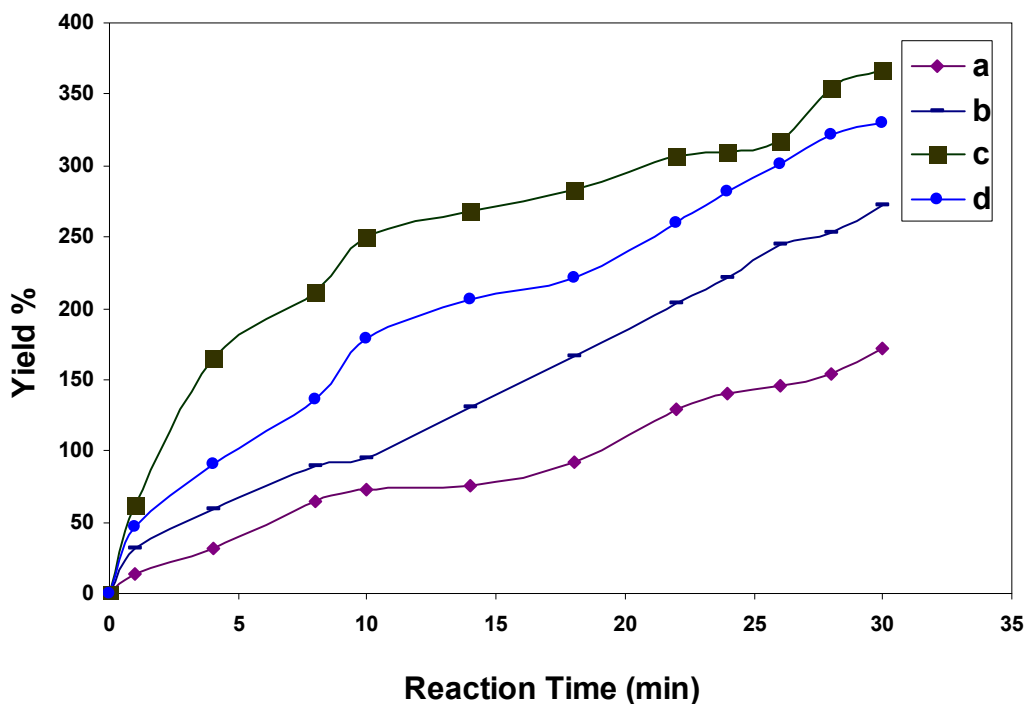


Fig.1. Reaction temperature dependence for the yield % at variable reduction and decomposition temperature. yield % at (a) 500 °C, (b) 550 °C, (c) 600 °C, (d) 650 °C.

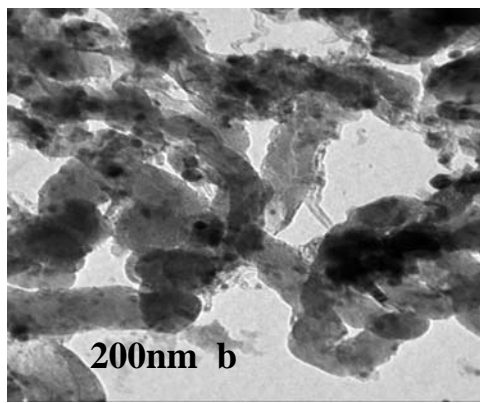
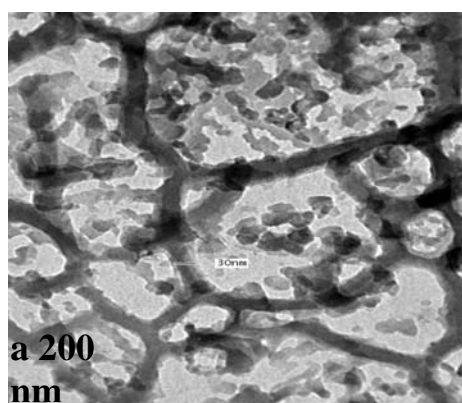


Fig.2. TEM images of CNTs produced by the decomposition of acetylene on SHF supported on alumina at the same reduction and decomposition temperature (a) 500 °C, (b) 600 °C.

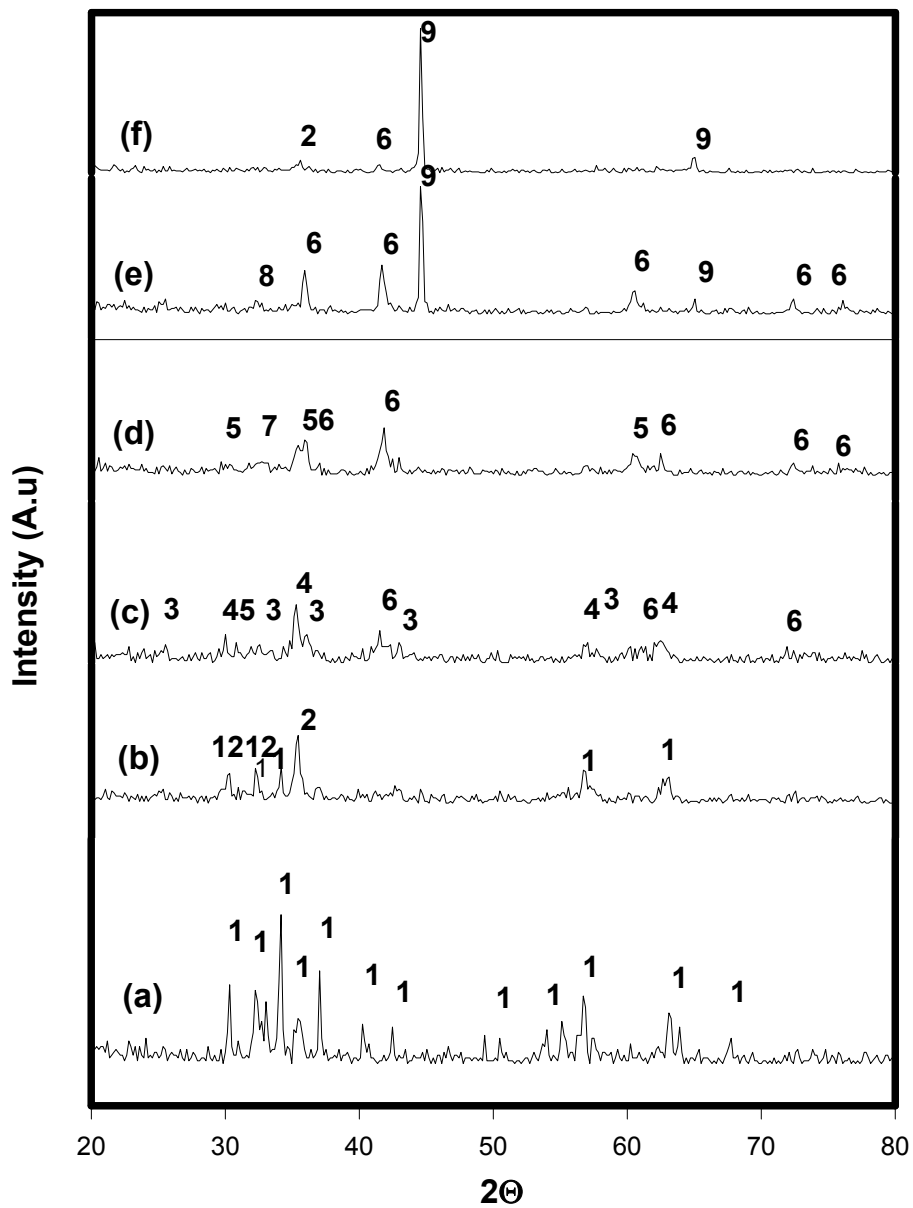


Fig.3. The XRD diffractograms of SHF compacts completely reduced at 400 to 600 °C in H<sub>2</sub> flow. (1) SrFe<sub>12</sub>O<sub>19</sub>, (2) Fe<sub>2</sub>O<sub>3</sub>, (3) Sr<sub>7</sub>Fe<sub>10</sub>O<sub>22</sub>, (4) Fe<sub>3</sub>O<sub>4</sub>, (5) Fe<sub>21.34</sub>O<sub>32</sub>, (6) FeO, (7) Sr<sub>4</sub>Fe<sub>6</sub>O<sub>13</sub>, (8)Sr<sub>2</sub>Fe<sub>2</sub>O<sub>5</sub>, (9)Fe.

(a) un reduced SHF, (b) SHF reduced at 400 °C, (c) SHF reduced at 450 °C,  
 (d) SHF reduced at 500 °C, (e) SHF reduced at 550 °C, (f) SHF reduced at 600 °C.

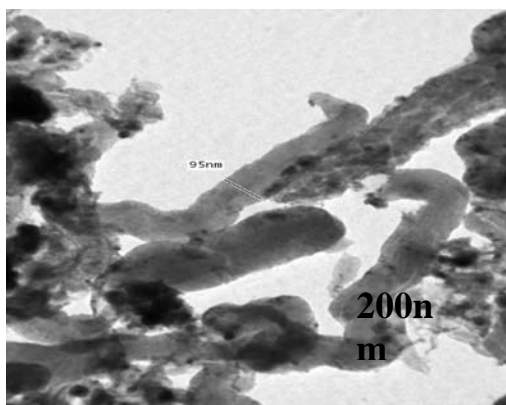


Fig.4. TEM images of CNTs produced on SHF reduced at 600 °C by the decomposition of acetylene at 600 °C.

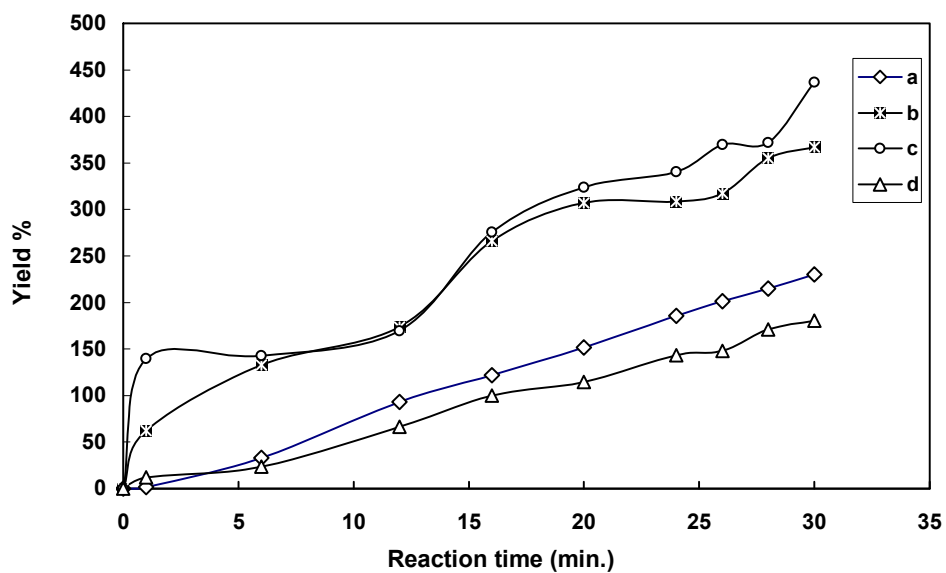


Fig.5. Reaction temperature dependence for the yield % at variable decomposition temperature and constant reduction temperature (600 °C)  
Yield % at (a) 500 °C, (b) 600 °C, (c) 700 °C, (d) 800 °C.

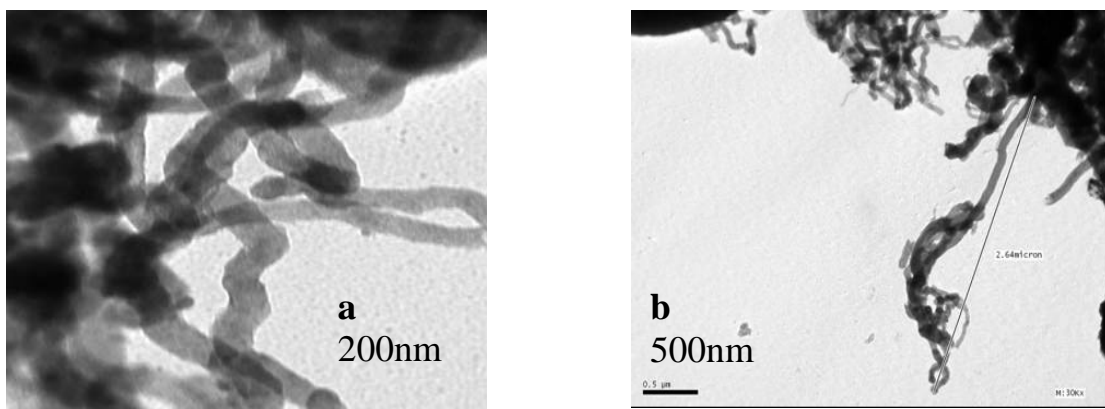


Fig.6. TEM images of CNTs produced on SHF reduced at 600 °C by the decomposition of acetylene at 500 °C.

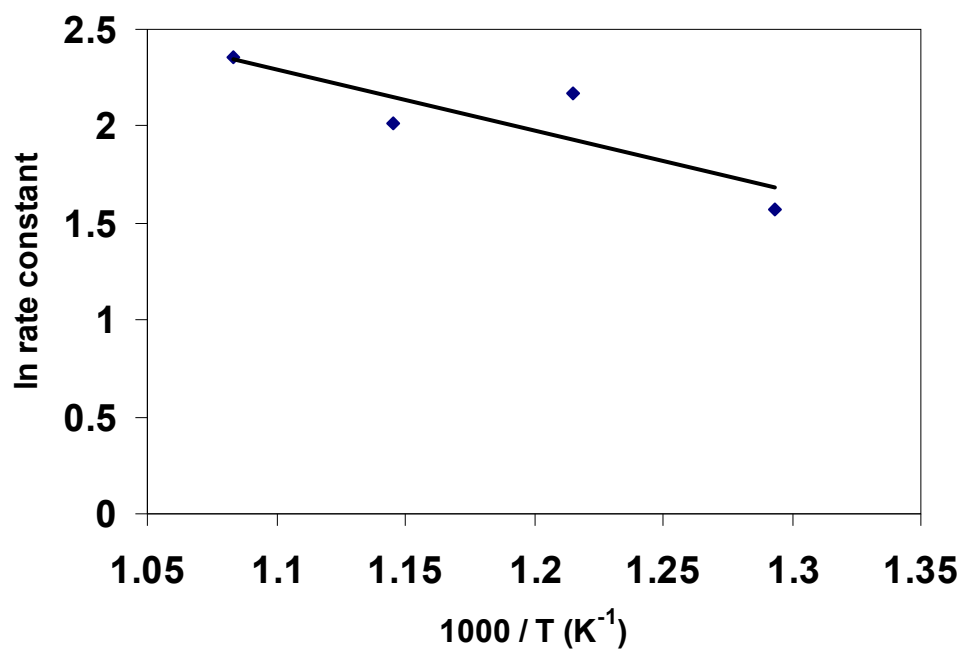


Fig.7. Arrhenius plot of CNTs synthesis on reduced SHF supported on alumina at different reduction temperature.

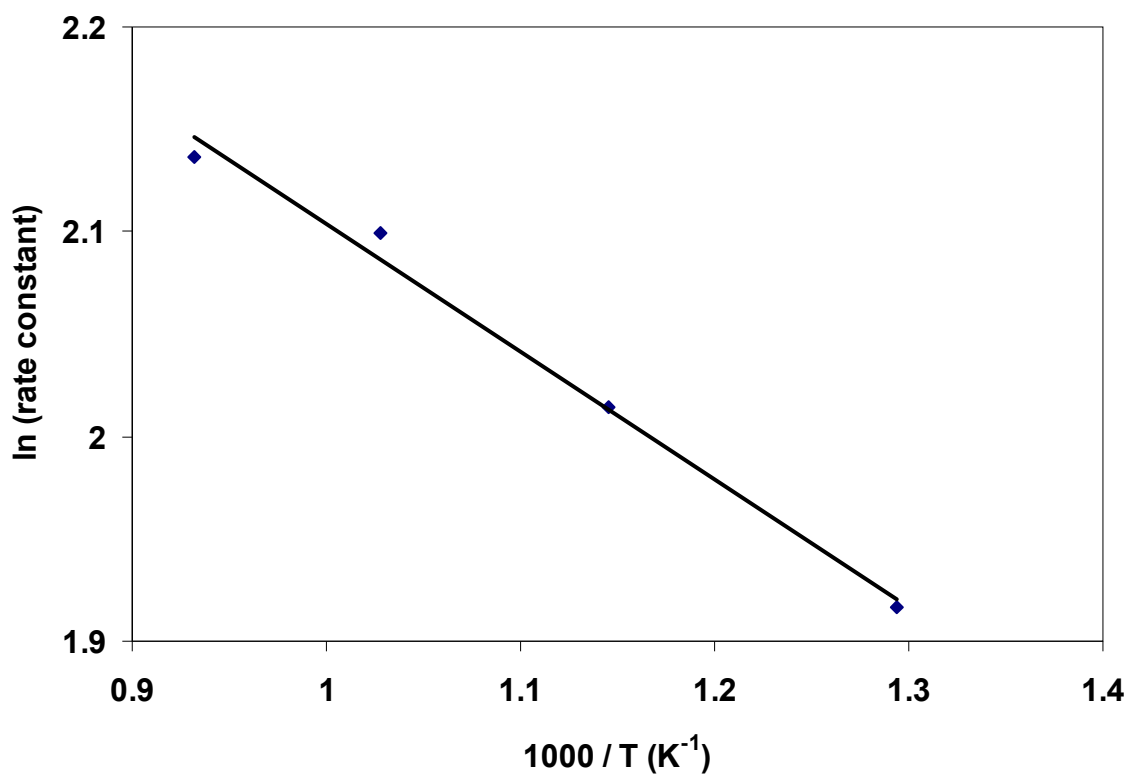


Fig.8. Arrhenius plot of CNTs synthesis on reduced SHF supported on alumina at constant reduction temperature.

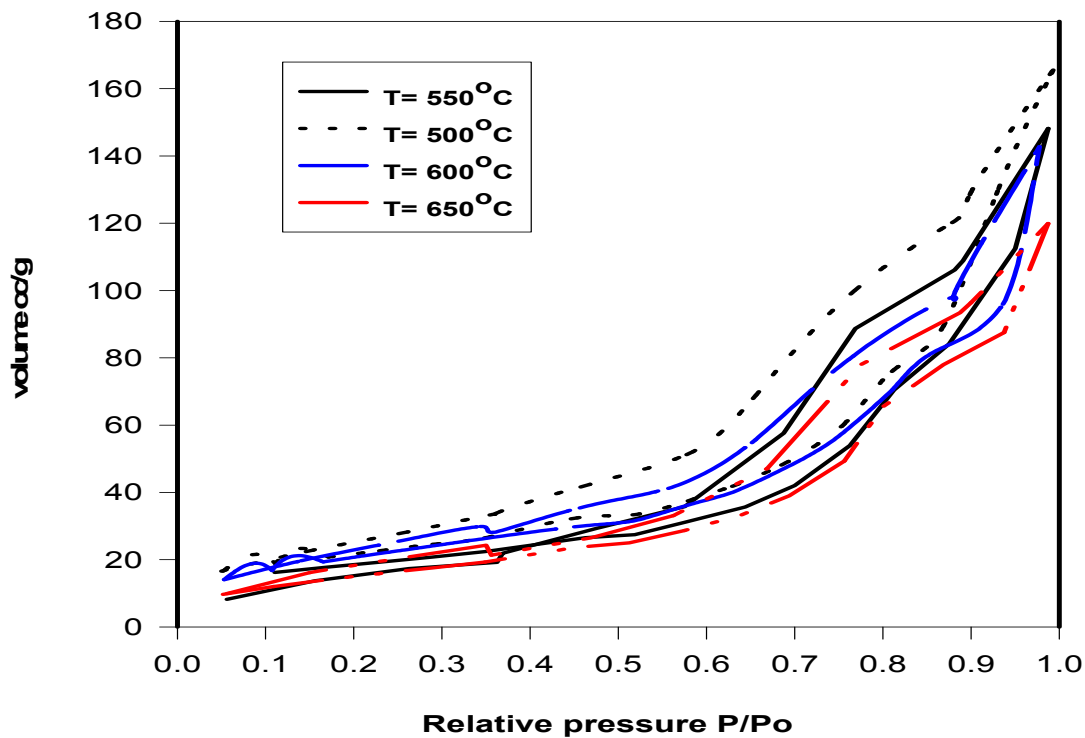


Fig.9. Hysteresis loops of Strontium hexaferrite ( $\text{SrFe}_{12}\text{O}_{19}$ ) supported on alumina with the molar ratio 40 SHF: 60 $\text{Al}_2\text{O}_3$  reduced at different temperature.

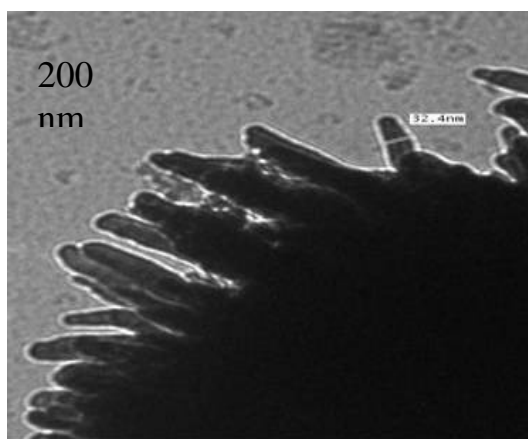


Fig.10. TEM image for SHF reduced at 700 °C