

## APPENDICES

### Appendix A

#### *F* Test

Table A-1 F Distribution critical values for 90% confidence

Denominator DF												
↓	Numerator DF											
	1	2	3	4	5	6	7	8	9	10	12	15
1	39.863	49.500	53.593	55.833	57.240	58.204	58.906	59.439	59.858	60.195	60.705	61.220
2	8.526	9.000	9.162	9.243	9.293	9.326	9.349	9.367	9.381	9.392	9.408	9.425
3	5.538	5.462	5.391	5.343	5.309	5.285	5.266	5.252	5.240	5.230	5.216	5.200
4	4.545	4.325	4.191	4.107	4.051	4.010	3.979	3.955	3.936	3.920	3.896	3.870
5	4.060	3.780	3.619	3.520	3.453	3.405	3.368	3.339	3.316	3.297	3.268	3.238
6	3.776	3.463	3.289	3.181	3.108	3.055	3.014	2.983	2.958	2.937	2.905	2.871
7	3.589	3.257	3.074	2.961	2.883	2.827	2.785	2.752	2.725	2.703	2.668	2.632
8	3.458	3.113	2.924	2.806	2.726	2.668	2.624	2.589	2.561	2.538	2.502	2.464
9	3.360	3.006	2.813	2.693	2.611	2.551	2.505	2.469	2.440	2.416	2.379	2.340
10	3.285	2.924	2.728	2.605	2.522	2.461	2.414	2.377	2.347	2.323	2.284	2.244
11	3.225	2.860	2.660	2.536	2.451	2.389	2.342	2.304	2.274	2.248	2.209	2.167
12	3.177	2.807	2.606	2.480	2.394	2.331	2.283	2.245	2.214	2.188	2.147	2.105
13	3.136	2.763	2.560	2.434	2.347	2.283	2.234	2.195	2.164	2.138	2.097	2.053
14	3.102	2.726	2.522	2.395	2.307	2.243	2.193	2.154	2.122	2.095	2.054	2.010
15	3.073	2.695	2.490	2.361	2.273	2.208	2.158	2.119	2.086	2.059	2.017	1.972

Taken from: <http://www.statsoft.com/textbook/sttable.html>

Table A-2 *F* Distribution critical values for 95% confidence

Denominator DF												
↓	Numerator DF											
	1	2	3	4	5	6	7	8	9	10	12	15
1	161.448	199.500	215.707	224.583	230.162	233.986	236.768	238.883	240.543	241.882	243.906	245.950
2	18.513	19.000	19.164	19.247	19.296	19.330	19.353	19.371	19.385	19.396	19.413	19.429
3	10.128	9.552	9.277	9.117	9.014	8.941	8.887	8.845	8.812	8.786	8.745	8.703
4	7.709	6.944	6.591	6.388	6.256	6.163	6.094	6.041	5.999	5.964	5.912	5.858
5	6.608	5.786	5.410	5.192	5.050	4.950	4.876	4.818	4.773	4.735	4.678	4.619
6	5.987	5.143	4.757	4.534	4.387	4.284	4.207	4.147	4.099	4.060	4.000	3.938
7	5.591	4.737	4.347	4.120	3.972	3.866	3.787	3.726	3.677	3.637	3.575	3.511
8	5.318	4.459	4.066	3.838	3.688	3.581	3.501	3.438	3.388	3.347	3.284	3.218
9	5.117	4.257	3.863	3.633	3.482	3.374	3.293	3.230	3.179	3.137	3.073	3.006
10	4.965	4.103	3.708	3.478	3.326	3.217	3.136	3.072	3.020	2.978	2.913	2.845
11	4.844	3.982	3.587	3.357	3.204	3.095	3.012	2.948	2.896	2.854	2.788	2.719
12	4.747	3.885	3.490	3.259	3.106	2.996	2.913	2.849	2.796	2.753	2.687	2.617
13	4.667	3.806	3.411	3.179	3.025	2.915	2.832	2.767	2.714	2.671	2.604	2.533
14	4.600	3.739	3.344	3.112	2.958	2.848	2.764	2.699	2.646	2.602	2.534	2.463
15	4.543	3.682	3.287	3.056	2.901	2.791	2.707	2.641	2.588	2.544	2.475	2.403

Taken from: <http://www.statsoft.com/textbook/sttable.html>

Table A-3 *F* Distribution critical values for 99% confidence

Denominator DF												
↓	Numerator DF											
	1	2	3	4	5	6	7	8	9	10	12	15
1	4052.18	4999.50	5403.35	5624.58	5763.65	5858.99	5928.36	5981.07	6022.47	6055.85	6106.32	6157.29
2	98.50	99.00	99.17	99.25	99.30	99.33	99.36	99.37	99.39	99.40	99.42	99.43
3	34.116	30.817	29.457	28.710	28.237	27.911	27.672	27.489	27.345	27.229	27.052	26.872
4	21.198	18.000	16.694	15.977	15.522	15.207	14.976	14.799	14.659	14.546	14.374	14.198
5	16.258	13.274	12.060	11.392	10.967	10.672	10.456	10.289	10.158	10.051	9.888	9.722
6	13.745	10.925	9.780	9.148	8.746	8.466	8.260	8.102	7.976	7.874	7.718	7.559
7	12.246	9.547	8.451	7.847	7.460	7.191	6.993	6.840	6.719	6.620	6.469	6.314
8	11.259	8.649	7.591	7.006	6.632	6.371	6.178	6.029	5.911	5.814	5.667	5.515
9	10.561	8.022	6.992	6.422	6.057	5.802	5.613	5.467	5.351	5.257	5.111	4.962
10	10.044	7.559	6.552	5.994	5.636	5.386	5.200	5.057	4.942	4.849	4.706	4.558
11	9.646	7.206	6.217	5.668	5.316	5.069	4.886	4.744	4.632	4.539	4.397	4.251
12	9.330	6.927	5.953	5.412	5.064	4.821	4.640	4.499	4.388	4.296	4.155	4.010
13	9.074	6.701	5.739	5.205	4.862	4.620	4.441	4.302	4.191	4.100	3.960	3.815
14	8.862	6.515	5.564	5.035	4.695	4.456	4.278	4.140	4.030	3.939	3.800	3.656
15	8.683	6.359	5.417	4.893	4.556	4.318	4.142	4.004	3.895	3.805	3.666	3.522

Taken from: <http://www.statsoft.com/textbook/sttable.html>

Table A-4 ANOVA result for GNF yield of samples FG

Source of variance	Degree of freedom ( <i>df</i> )	Sum of squares ( <i>S</i> )	Variance ( <i>V</i> )	Variance ratio ( <i>F</i> )	Percent contribution (%)
Time	2	8.831	4.415	1.911	1.753
C <sub>2</sub> H <sub>4</sub> /H <sub>2</sub> flow	(2)	(2.563)	<i>(pooled)</i>		
Temperature	2	222.102	111.051	48.064	90.551
Error	4	9.241	2.31		7.696
Total	8	240.175			100

Table A-5 ANOVA result for GNF yield of samples NG

Source of variance	Degree of freedom ( <i>df</i> )	Sum of squares ( <i>S</i> )	Variance ( <i>V</i> )	Variance ratio ( <i>F</i> )	Percent contribution (%)
Time	(2)	(0.063)	<i>(pooled)</i>		
C <sub>2</sub> H <sub>4</sub> /H <sub>2</sub> flow	2	11.749	27.539	11.322	63.741
Temperature	2	5.16	12.096	4.734	26.651
Error	4	0.853			9.608
Total	8				100

Table A-6 ANOVA result for GNF yield of samples LFe

Source of variance	Degree of freedom ( <i>df</i> )	Sum of squares ( <i>S</i> )	Variance ( <i>V</i> )	Variance ratio ( <i>F</i> )	Percent contribution (%)
Time	2	14.275	7.137	9.79	1.807
C <sub>2</sub> H <sub>4</sub> /H <sub>2</sub> flow	2	41.942	20.971	28.764	5.708
Temperature	2	651.488	325.744	446.795	91.661
Error	2	1.457	0.728		0.824
Total	8	709.164			100

Table A-7 ANOVA result for GNF yield of samples LNi

Source of variance	Degree of freedom ( <i>df</i> )	Sum of squares ( <i>S</i> )	Variance ( <i>V</i> )	Variance ratio ( <i>F</i> )	Percent contribution (%)
Time	2	50.656	25.328	8.306	54.631
C <sub>2</sub> H <sub>4</sub> /H <sub>2</sub> flow	(2)	(5.874)	<i>(pooled)</i>		
Temperature	2	18.707	9.353	3.067	15.459
Error	4	12.196	3.049		29.91
Total	8	81.56			100

## Appendix B

### Analytical equipment for material characterization

The basic steps for all electron microscopes are as below (Central Facility for Electron Microscopy (CFEM), 2003):

- a. By using a positive electrical potential, electrons are formed and then accelerated towards the specimen.
- b. By using metal apertures and magnetic lenses, the electron stream is confined into thin, focused monochromatic beam.
- c. With a magnetic lens the beam is focused onto a sample.
- d. The effects due to interaction inside the irradiated sample are detected and later are transformed into an image.

#### B-1 Scanning electron microscope

The main features of SEM include the electron gun, the magnetic focusing lenses, the sample vacuum chamber and stage region as well as the electronics console containing the control panel, the electronic power supplies and the scanning modules. Usually a solid state energy dispersive X-ray (EDS) detector is attached to the column as an additional tool for characterization (Bindell, 1992).

The working principles of SEM can be based from the electron optics in Figure B-1. In this figure, the virtual source represents the electron gun which produces the monochromatic electrons. There are three types of electron source: thermionic tungsten, Lanthanum Hexaboride ( $\text{LaB}_6$ ), and hot and cold field emission (Bindell, 1992). The electron stream is first condensed by the first condenser lens which limits the amount of current in the beam. In conjunction with the condenser aperture, it eliminates the high-angle electrons from the beam. Next, the second condenser lens forms the electrons into a thin, tight, coherent beam before the objective aperture further eliminates high-angle electrons from the beam. Later, the scan coils sweep the beam in a grid fashion and dwell on points for a certain time as determined by the scan speed. The final lens or objective lens then focuses the scanning beam onto parts of specimen desired. When the beam strikes the specimen, interactions occur

inside the sample. These interactions are detected with various instruments. The instrument counts the number of interactions and displays a pixel on a cathode ray tube (CRT). After that the beam moves to its next dwell points. The process is repeated until the grid scan is completed (Central Facility for Electron Microscopy (CFEM), 2003). Today, SEM comes in different versions such as virtual SEM (VSEM), environmental SEM (ESEM) and field emission (FESEM) which has higher resolution.

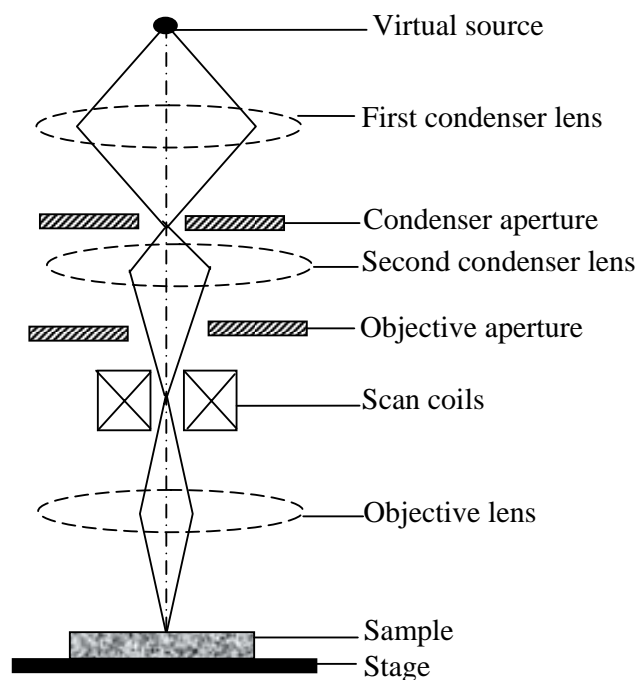


Figure B-1 Schematic of electron optics in SEM (Central Facility for Electron Microscopy (CFEM), 2003)

## B-2 Transmission electron microscope

In TEM, the ray of electrons is produced by a pin-shaped cathode heated up by current. The electrons are vacuumed up by a high voltage at the anode with acceleration voltage between 50 and 150 kV. The higher the voltage, the shorter are the electron waves and the higher is the power of resolution (Sengbusch, 2003). The beam of electrons is then focused by the first and second condenser lenses to obtain a small, thin, coherent beam (refer to Figure B-2). The first lens is controlled by the

"spot size knob" in order to determine the "spot size" while the second lens is controlled by the "intensity or brightness knob" that is to change the size of the spot on the sample i.e. from a wide dispersed spot to a pinpoint beam (Central Facility for Electron Microscopy (CFEM), 2003).

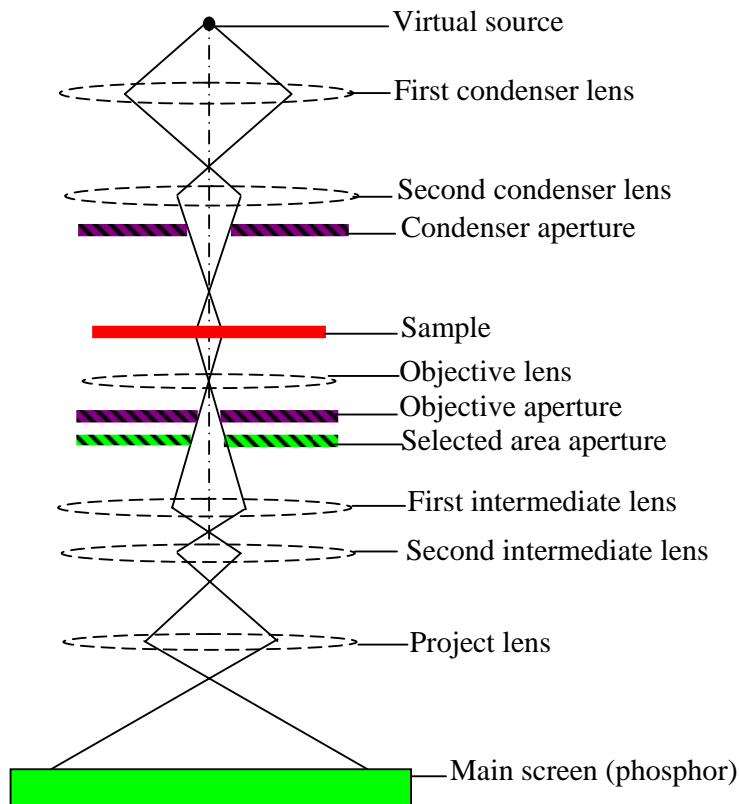


Figure B-2 Schematic of electron optics in TEM (Central Facility for Electron Microscopy, 2003)

After passing through the second condenser lens, the beam is restricted by the condenser aperture just before it strikes the specimen. Here parts of the beam are transmitted and this transmitted portion is focused by the objective lens into an image. The image is enhanced by objective aperture which blocks out high-angle diffracted electrons while selected area aperture enables the user to examine the periodic diffraction of electrons by ordered arrangements of atoms in the sample (Central Facility for Electron Microscopy, 2003). Finally, the image strikes the phosphor image screen and light is generated to allow the user to see the image. The darker regions of the image represent those areas of the sample where more

electrons are scattered (denser or thicker regions in specimen) whereas the lighter regions represent those areas of the sample that more electrons are transmitted through (thinner regions in specimen) (Davis, 2003).

### B-3 Raman spectroscopy

There are four major components in Raman spectroscopy. These consist of:

- Excitation source that is a continuous wave (CW) laser
- Sample illumination and collection system
- Wavelength selector that is spectrograph/monochromator
- Detection and computer control that is CCD

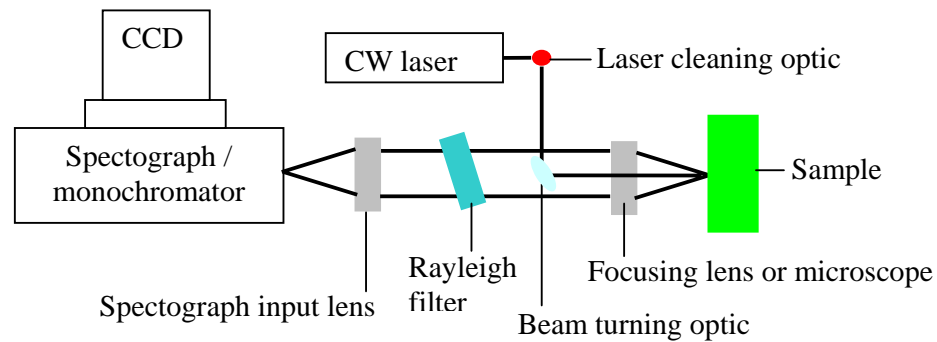


Figure B-3 Schematic diagram of Raman spectroscopy set up (Andor Technology, 2009)

Based from Figure B-3, a sample is illuminated with a laser beam originated from a continuous wave (CW) laser. The light from illuminated spot is collected with a lens and sent through a spectrograph or monochromator. However, wavelengths that are close to the laser line are filtered out by Rayleigh filter while the rest of the collected light will be dispersed onto a detector. The detection system consists of dispersive monochromator with a charged couple device (CCD) detector (Ferraro, Nakamoto, & Brown, 2003). CW lasers such as Ar<sup>+</sup> (wavelength between 351.1 – 514.5 nm), Kr<sup>+</sup> (wavelength between 337.4 – 676.4 nm) and He-Ne (wavelength 632.8 nm) are commonly used in Raman spectroscopy. Recently, pulsed lasers such as Nd:YAG (neodymium-doped yttrium aluminium garnet), diode and excimer lasers have been used for time resolved and ultra-violet (UV) resonance Raman spectroscopy (Ferraro et al., 2003). Because Raman scattering is a weak



process, a key requirement to obtain Raman spectra is that the spectrometer provides a high rejection of scattered laser light. New methods such as very narrow rejection filters and Fourier-transform techniques are becoming more widespread (Tissue, 1996).

#### B-4 X-ray diffractometer

About 95% of all solid materials can be described as crystalline where they exist in crystal form. A crystal is known to possess long-range order because it is composed of atoms arranged in a regular ordered pattern in three dimensions (Suryanarayana & Grant Norton, 1998). When X-rays interact with a crystalline substance, a diffraction pattern is obtained (Scintag Inc., 1999). This can be seen in Figure B-4.

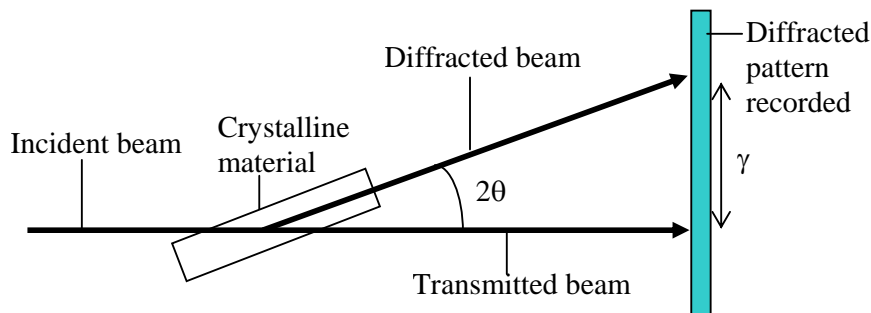


Figure A-4 Diffraction of x-ray by a crystalline material (Moeck, 2004)

The three basic components of XRD are x-ray source, specimen and x-ray detector. These components lie on the circumference of a circle known as focusing circle (Suryanarayana & Grant Norton, 1998) (refer to Figure B-5). The angle between the specimen plane and the x-ray source is known as Bragg's angle,  $\theta$  while the angle between the projection of the x-ray source and the detector is known as  $2\theta$ . The radius of the focusing circle is increases as the angle  $2\theta$  decreases. The X-ray radiation most commonly used is that emitted by copper (Cu), whose characteristic wavelength for the Cu  $K\alpha$  radiation is  $1.5418\text{\AA}$  (Whittingham, 1997)

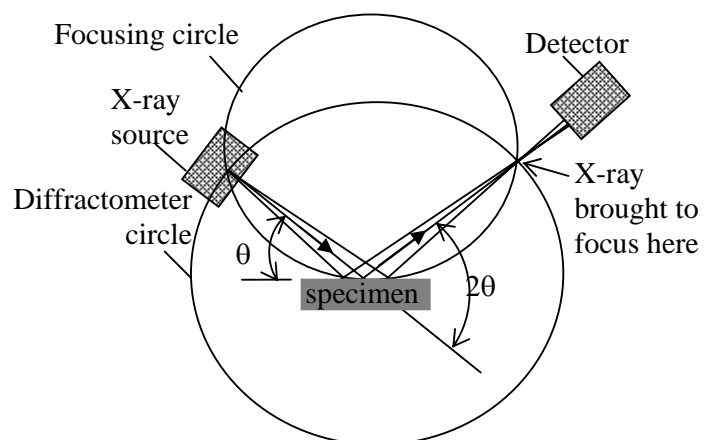


Figure B-5 Geometry of an x-ray diffractometer (Suryanarayana & Grant Norton, 1998)

### Appendix C

#### Experimental and analysis data of the synthesized catalyst, CNTs and GNFs

Table C-1 Catalyst yield (mol%)

Sample	Yield 1	Yield 2	Average
Fe1	98.48	94.45	96.5
Fe2	85.6	90.06	87.8
Fe3	95.23	93.97	94.6
Fe4	91.84	92.09	92.0
Fe5	94.08	86.74	90.4
Fe6	92.76	84.33	88.5
Fe7	77.47	62.89	70.2
Fe8	89	84.33	86.7
Fe9	81.79	73.87	77.8

Sample	Yield 1	Yield 2	Average
Ni1	83.42	84.36	83.9
Ni2	66.86	90.33	78.6
Ni3	86.27	88.44	87.4
Ni4	72.94	84.1	78.5
Ni5	92.5	90.66	91.6
Ni6	76.96	68.78	72.9
Ni7	84.8	86.21	85.5
Ni8	79.1	83.06	81.1
Ni9	78.62	84.88	81.8

Table C-2 Reaction yield of GNFs (g/(g<sub>cat</sub>·hr))

Sample	Yield 1	Yield 2	Average
1FG	1.074	1.656	1.365
2FG	3.713	4.524	4.119
3FG	2.009	2.529	2.269
4FG	6.590	6.269	6.430
5FG	2.600	2.206	2.403
6FG	1.052	1.105	1.079
7FG	1.847	1.631	1.739
8FG	1.068	1.107	1.088
9FG	3.008	5.971	4.490
1NG	23.948	28.779	26.364
2NG	32.140	28.564	30.352
3NG	20.110	22.947	21.529
4NG	28.842	36.915	32.879
5NG	25.011	26.631	25.821
6NG	19.823	20.786	20.305
7NG	32.956	31.827	32.391
8.NG	24.063	22.367	23.215
9NG	25.100	22.750	23.925

Sample	Yield 1	Yield 2	Average
L <sub>1</sub> Fe	1.660	1.860	1.760
L <sub>2</sub> Fe	3.062	1.967	2.514
L <sub>3</sub> Fe	9.829	10.631	10.230
L <sub>4</sub> Fe	1.660	2.930	2.295
L <sub>5</sub> Fe	12.280	11.300	11.790
L <sub>6</sub> Fe	0.860	0.833	0.847
L <sub>7</sub> Fe	12.758	11.370	12.064
L <sub>8</sub> Fe	1.267	1.352	1.309
L <sub>9</sub> Fe	0.906	0.984	0.945
L <sub>1</sub> Ni	51.522	50.676	51.099
L <sub>2</sub> Ni	75.707	79.505	77.606
L <sub>3</sub> Ni	66.573	68.329	67.451
L <sub>4</sub> Ni	48.284	47.100	47.692
L <sub>5</sub> Ni	35.934	28.027	31.981
L <sub>6</sub> Ni	35.654	37.069	36.361
L <sub>7</sub> Ni	43.658	45.650	44.654
L <sub>8</sub> Ni	23.755	23.575	23.665
L <sub>9</sub> Ni	37.739	42.008	39.873

Table C-3 CNTs specific BET surface area (m<sup>2</sup>/g) and true density (g/cm<sup>3</sup>)

Sample	Surface area	Density
1R	92.44	2.359
2R	120.80	2.951
3R	64.70	2.703
4R	60.40	2.501
5R	68.99	3.051

Sample	Surface area	Density
6R	70.24	2.623
7R	50.90	2.471
8R	60.70	2.680
9R	56.92	2.590

Table C-4 GNFs specific BET surface area ( $\text{m}^2/\text{g}$ ) and true density ( $\text{g}/\text{cm}^3$ )

Preliminary experiment			Modified experiment		
Sample	Surface area	Density	Sample	Surface area	Density
1FG	180.15	2.446	L1Fe	127.5	2.624
2FG	256.80	2.015	L2Fe	111.3	2.983
3FG	83.88	2.283	L3Fe	92.56	2.290
4FG	259.05	2.274	L4Fe	195.6	2.301
5FG	94.47	2.719	L5Fe	147.09	2.203
6FG	135.30	2.508	L6Fe	88.08	2.520
7FG	87.52	2.549	L7Fe	146.54	2.087
8FG	211.40	2.481	L8Fe	85.39	2.601
9FG	293	2.781	L9Fe	98.31	2.589
1NG	230.80	1.809	L1Ni	291.7	1.927
2NG	215.40	2.004	L2Ni	135.7	1.891
3NG	165.10	2.213	L3Ni	196.9	2.076
4NG	249.90	1.951	L4Ni	139.1	2.148
5NG	155.10	2.144	L5Ni	61.7	2.600
6NG	281.20	2.216	L6Ni	110.95	1.940
7NG	132.60	2.347	L7Ni	60.44	2.237
8NG	252.30	1.928	L8Ni	200.28	1.965
9NG	199.10	1.994	L9Ni	111.54	1.932

Table C-5 X-ray diffraction carbon peaks for CNTs and GNFs

Samples	$2\theta$ (deg)	$d_{002}$ spacing ( $\text{\AA}$ )	Samples	$2\theta$ (deg)	$d_{002}$ spacing ( $\text{\AA}$ )	Samples	$2\theta$ (deg)	$d_{002}$ spacing ( $\text{\AA}$ )
1R	26.31	3.384	8FG	26.20	3.40	L4Fe	23.36	3.40
2R	26.26	3.39	9FG	26.01	3.42	L5Fe	23.60	3.40
3R	26.24	3.39				L6Fe	25.12	3.43
4R	26.30	3.39	1NG	26.29	3.39	L7Fe	22.96	3.39
5R	26.31	3.39	2NG	26.31	3.38	L8Fe	24.56	3.42
6R	26.34	3.38	3NG	26.26	3.39	L9Fe	25.20	3.38
7R	26.29	3.39	4NG	26.24	3.39			
8R	26.38	3.38	5NG	26.30	3.39	L1Ni	25.94	3.43
9R	26.32	3.38	6NG	26.31	3.38	L2Ni	26.10	3.41
1FG	26.54	3.36	7NG	26.34	3.38	L3Ni	26.07	3.41
2FG	26.24	3.39	8NG	26.29	3.39	L4Ni	25.66	3.47
3FG	26.38	3.38	9NG	26.38	3.38	L5Ni	26.34	3.38
4FG	26.02	3.42				L6Ni	25.87	3.44
5FG	26.39	3.37	L1Fe	24.56	3.40	L7Ni	26.21	3.40
6FG	26.33	3.38	L2Fe	25.12	3.38	L8Ni	26.74	3.33
7FG	26.35	3.38	L3Fe	24.80	3.39	L9Ni	25.90	3.44

Table C-6 Raman spectra bands of CNTs and GNFs ( $\text{cm}^{-1}$ )

Sample	Amorphous ( $I_A$ ) $1500 \text{ cm}^{-1}$	D band intensity ( $I_D$ ) $1330\text{-}1360 \text{ cm}^{-1}$	G band intensity ( $I_G$ ) $1560\text{-}1600 \text{ cm}^{-1}$	Relative value of amorphous to graphite	Degree of graphitization
1R	5.602	9.383	17.725	0.316	0.654
2R	4.423	3.916	8.299	0.533	0.679
3R	5.433	6.268	8.557	0.635	0.577
4R	15.273	16.674	25.344	0.603	0.603
5R	7.357	11.442	28.563	0.258	0.714
6R	3.100	5.135	9.042	0.343	0.638
7R	3.466	3.854	7.762	0.447	0.668
8R	5.051	6.885	20.662	0.244	0.750
9R	13.712	15.487	16.475	0.832	0.515
1FG	8.027	13.417	11.265	0.713	0.456
2FG	10.233	15.418	14.518	0.705	0.485
3FG	11.001	15.403	27.048	0.363	0.407
4FG	7.602	10.238	9.673	0.786	0.486
5FG	10.439	18.826	32.377	0.368	0.309
6FG	8.805	9.347	12.131	0.435	0.726
7FG	7.376	18.624	28.787	0.256	0.607
8FG	8.616	10.202	10.699	0.805	0.512
9FG	7.600	10.749	11.277	0.674	0.512
1NG	34.876	28.103	43.120	0.652	0.553
2NG	12.146	10.752	11.631	0.924	0.489
3NG	16.021	9.983	13.892	0.719	0.464
4NG	12.740	9.203	12.199	0.754	0.489
5NG	28.539	8.346	20.518	0.407	0.418
6NG	10.705	7.658	10.449	0.733	0.494
7NG	11.702	8.896	10.727	0.829	0.478
8NG	12.996	10.445	12.837	0.814	0.497
9NG	12.525	10.272	12.399	0.829	0.498
L1Fe	9.505	12.382	12.550	0.757	0.503
L2Fe	15.561	34.297	39.890	0.390	0.538
L3Fe	11.115	19.627	26.691	0.416	0.576
L4Fe	14.984	22.023	22.830	0.656	0.509
L5Fe	9.597	15.750	16.230	0.591	0.508
L6Fe	9.894	11.579	11.821	0.837	0.505
L7Fe	9.999	18.688	21.011	0.476	0.529
L8Fe	8.653	10.723	10.929	0.792	0.505
L9Fe	13.193	18.325	19.773	0.667	0.519
L1Ni	10.824	13.793	14.080	0.769	0.505
L2Ni	11.600	14.044	14.608	0.794	0.510
L3Ni	12.100	14.962	15.073	0.803	0.502
L4Ni	11.419	14.697	13.843	0.825	0.485
L5Ni	10.735	15.893	16.277	0.660	0.506
L6Ni	11.008	14.376	14.330	0.768	0.499
L7Ni	21.082	33.668	33.965	0.621	0.502
L8Ni	15.782	21.648	21.773	0.725	0.501
L9Ni	13.437	17.156	18.488	0.727	0.519

Note: Relative value of amorphous to graphite =  $I_A/I_G$

Degree of graphitization =  $I_G/(I_D+I_G)$

## Appendix D

### Hydrogen adsorption data

Table D-1 Hydrogen adsorption experimental data at 77 K

	Adsorption		Desorption			Adsorption		Desorption		
	Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)		Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)	
Sample 9FG	0.00	0.1103	19.68	0.6427	Commercial SWNT	0.00	0.1490	20.10	1.0675	
	0.03	0.0484	16.01	0.6280		0.02	0.0560	20.03	1.0693	
	0.30	0.1894	13.55	0.6112		0.30	0.2602	16.07	1.0257	
	1.02	0.3023	10.95	0.5886		1.02	0.4638	13.57	0.9914	
	2.50	0.4120	8.94	0.5652		2.52	0.6382	10.95	0.9454	
	4.02	0.4672	7.81	0.5497		4.04	0.7326	8.73	0.8983	
	5.53	0.5040	6.88	0.5345		5.52	0.7941	8.00	0.8807	
	7.07	0.5311	5.89	0.5150		7.06	0.8437	7.01	0.8536	
	8.59	0.5593	4.91	0.4916		8.53	0.8820	5.97	0.8215	
	9.94	0.5721	4.00	0.4647		10.08	0.9178	4.90	0.7815	
	11.67	0.5951	2.41	0.3993		11.58	0.9508	4.01	0.7406	
	13.16	0.6043	1.40	0.3245		12.88	0.9694	2.49	0.6456	
	14.12	0.6108	0.97	0.2667		14.18	0.9905	1.36	0.5266	
	15.15	0.6174	0.50	0.1751		15.14	1.0048	0.97	0.4606	
	16.61	0.6256	0.10	0.0516		16.61	1.0230	0.48	0.3376	
	18.14	0.6340	0.01	0.0293		18.11	1.0429	0.09	0.1433	
	20.14	0.6423				20.10	1.0675	0.01	0.0912	
	Sample 4NG	0.02	0.0012	20.11		0.5809				
		0.30	0.1230	20.01		0.5842				
1.02		0.2259	16.03	0.5571						
2.54		0.3186	13.55	0.5339						
4.05		0.3717	11.04	0.5070						
5.55		0.4094	9.02	0.4802						
7.06		0.4395	7.99	0.4646						
8.55		0.4649	6.96	0.4468						
10.08		0.4878	6.00	0.4277						
11.96		0.5041	4.90	0.4032						
13.15		0.5174	3.97	0.3783						
14.10		0.5250	2.50	0.3265						
15.13		0.5371	1.39	0.2651						
16.59		0.5520	0.98	0.2317						
18.10		0.5635	0.47	0.1665						
20.11	0.5809	0.09	0.0643							
		0.01	0.0206							

Table D-2 Hydrogen adsorption experimental data at 298 K

	Adsorption		Desorption			Adsorption		Desorption	
	Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)		Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)
Sample 9FG	0	0.0000	137.2	0.2957	Sample L7Ni	0	0.0000	150.18	0.0938
	10.05	0.0941	130.85	0.2918		1.03	0.0029	140.00	0.0914
	19.97	0.1196	120.03	0.2830		2.8	0.0062	130.02	0.0863
	30.03	0.1404	109.97	0.2659		3.96	0.0066	120.08	0.0823
	39.98	0.1609	100.1	0.2517		4.97	0.0078	110.06	0.0779
	49.98	0.1786	90.12	0.2412		6.04	0.0085	100.27	0.0747
	59.99	0.1984	80.04	0.2294		6.94	0.0102	90.12	0.0693
	70.03	0.2120	70.23	0.2081		7.98	0.0106	79.99	0.0632
	80.04	0.2259	59.76	0.2003		9.07	0.0104	69.99	0.0581
	89.96	0.2356	49.99	0.1771		9.90	0.0120	59.91	0.0525
	100.04	0.2523	40.01	0.1551		19.94	0.0211	49.91	0.0447
	109.98	0.2634	30.01	0.1400		30.35	0.0278	40.00	0.0381
	120.11	0.2785	20.00	0.1179		39.97	0.0380	29.91	0.0299
	130.51	0.2911	9.98	0.0858		49.82	0.0444	19.88	0.0210
	137.17	0.2939	8.91	0.0791		59.97	0.0507	9.98	0.0125
	Sample L6Ni	0.00	0.0000	89.67		0.0662	69.99	0.0565	9.00
1.99		0.0344	79.65	0.0678	79.70	0.0641	7.98	0.0109	
3.99		0.0385	69.69	0.0646	90.04	0.0683	6.99	0.0083	
5.99		0.0381	59.68	0.0638	100.04	0.0728	6.00	0.0070	
10.00		0.0324	49.67	0.0636	110.04	0.0766	4.96	0.0051	
19.95		0.0395	39.68	0.0556	120.09	0.0827	3.96	0.0047	
39.68		0.0500	29.63	0.0534	130.01	0.0862	2.98	0.0029	
49.68		0.0612	19.57	0.0462	139.99	0.0897	1.96	0.0012	
59.70		0.0549	10.00	0.0389	150.18	0.0938			
69.73		0.0601	8.00	0.0417	Sample L7Fe	0.00	0.0000	89.70	0.1800
79.68		0.0574	5.99	0.0420		0.99	0.0425	79.69	0.1738
89.70		0.0591	3.99	0.0227		5.00	0.0579	69.73	0.1661
99.65		0.0665				10.00	0.0731	59.72	0.1515
0.00		0.0000				19.95	0.0985	49.67	0.1380
						29.62	0.1113	39.71	0.2404
						39.67	0.1362	29.65	0.1016
				49.66		0.1530	19.58	0.0793	
				59.66		0.1620	0.99	0.0182	
				69.68		0.1743	89.70	0.1800	
				79.64		0.1846	79.69	0.1738	
				89.67		0.1943	69.73	0.1661	
				99.65		0.2037	59.72	0.1515	

Table D-2 Hydrogen adsorption experimental data at 298 K (continued)

	Adsorption		Desorption			Adsorption		Desorption	
	Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)		Pressure (bar)	Uptake (wt%)	Pressure (bar)	Uptake (wt%)
Sample 4NG	0.00	0.0000	149.99	0.0567	Sample L5Fe	1.99	0.0322	89.66	0.1226
	2.66	0.0017	140.00	0.0556		3.99	0.0454	79.65	0.1136
	5.00	0.0050	130.03	0.0542		6.00	0.0531	69.70	0.1101
	9.00	0.0078	120.11	0.0525		8.00	0.0484	59.69	0.1053
	9.86	0.0080	110.03	0.0508		10.00	0.0547	49.68	0.0944
	20.02	0.0133	99.98	0.0476		19.95	0.0680	39.69	0.0857
	30.00	0.0176	90.12	0.0449		29.61	0.0828	29.64	0.0782
	39.90	0.0246	80.00	0.0409		39.66	0.0959	19.57	0.0659
	49.98	0.0280	70.04	0.0369		44.49	0.0970	10.00	0.0506
	59.99	0.0321	60.32	0.0325		49.65	0.1043	8.01	0.0499
	69.92	0.0367	49.99	0.0284		59.65	0.1081	6.00	0.0462
	80.01	0.0407	39.99	0.0247		69.68	0.1134	3.99	0.0389
	90.10	0.0447	29.94	0.0179		79.63	0.1227	1.99	0.0396
	100.00	0.0476	20.01	0.0134		89.65	0.1296		
	110.00	0.0506	10.00	0.0089		99.62	0.1315		
	120.15	0.0517	9.00	0.0086					
	129.99	0.0539							
	140.00	0.0554							
149.99	0.0567								



## Appendix E

### Prediction of hydrogen isotherm using Neuro-fuzzy system

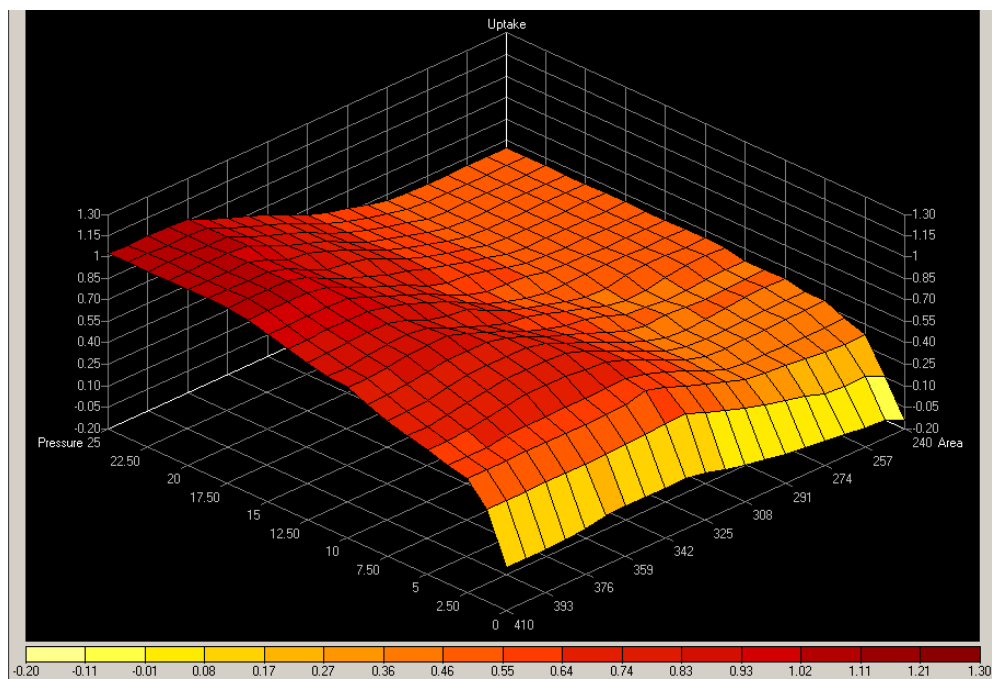


Figure E-1 3-D plot of hydrogen isotherm at 77 K and 20 bar.

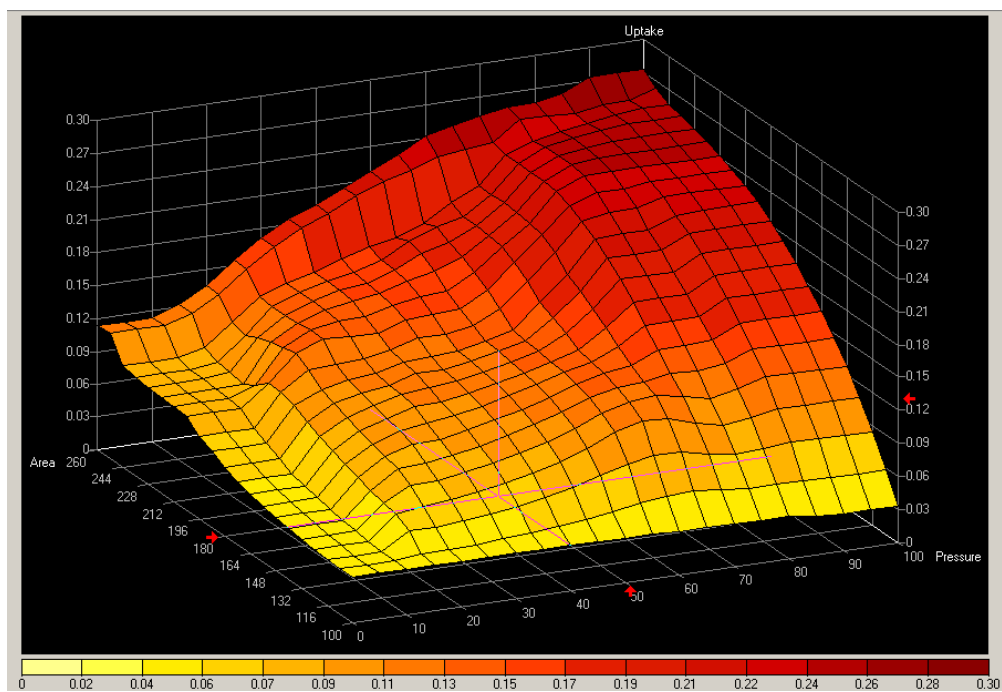


Figure E-2 3-D plot of hydrogen isotherm at 298 K and 100 bar.

## Appendix F

### List of publications, presentations and research participations

#### Journal Publications

1. S. Sufian, S. Yusup, G. S. Walker and A. M. Shariff, *Synthesis of graphitic nanofibres using iron (III) oxide catalyst for hydrogen storage application*, Materials Research Innovations, Volume 13, Issue 3, September Special Issue, 2009, pg 153-156.
2. S. Sufian, S. Yusup, G. Walker, T. Murugesan, *Synthesis and characterization of nanosized Fe<sub>2</sub>O<sub>3</sub> and NiO at different temperature by precipitation method*, Volume 45, Issue 3, June 2009, Indian Ceramic Review.
3. Suriati Sufian, Suzana Yusup, *Optimization for Development of Carbon Nanotubes using Taguchi Method at Constant Temperature*. Accepted by Journal of Nano Research.
4. Suriati Sufian, Suzana Yusup, Azmi M Shariff, *Hydrogen adsorption of graphitic - nanofibers at high pressure using magnetic suspension balance*. In review by Journal of Applied Science.

#### National conference

1. Suriati Sufian, *Synthesis of Nanosized Fe<sub>2</sub>O<sub>3</sub> and NiO Catalyst by Percipitation Method*, National Postgraduate Conference on Engineering, Science and Technology (NPC) 31 March 2008, UTP, Tronoh.

#### International Conferences

1. Suriati Sufian, Suzana Yusup, *Hydrogen Adsorption Model Using Neuro-fuzzy System*, Proceedings of the 5th WSEAS International Conference on Data Networks, Communications & Computers, 16-17 October, 2006, Bucharest, Romania.
2. S. Sufian, S. Yusup, G. S. Walker and A. M. Shariff, *Synthesis of graphitic nanofibres using iron (III) oxide catalyst for hydrogen storage application*, International Conference on Functional Material and Devices 2008 (ICFMD), 16-19 June 2008, KL.

3. Suriati Sufian, Suzana Yusup, *Synthesis and Characterization of Graphitic Nanofibres on Unsupported NiO*, The Trinidad and Tobago Gas Conference (TGTC), 7-10 October 2008, Tobago, Republic of Trinidad & Tobago.
4. Suriati Sufian, Suzana Yusup, *Optimization for Development of Carbon Nanotubes using Taguchi Method at Constant Temperature*, The 5<sup>th</sup> International Conference on Diffusion in Solids and Liquids (DSL 2009), 24-26 June 2009, Rome, Italy.
5. Suriati Sufian, Suzana Yusup, Azmi M Shariff, *Hydrogen adsorption of graphitic nanofibers at high pressure using magnetic suspension balance*, 3<sup>rd</sup> International Conference On Chemical & Bioprocess Engineering (ICCBPE-2009) - 23<sup>rd</sup> Symposium Of Malaysian Chemical Engineers (SOMChE-2009), 12-14 August 2009, Kota Kinabalu, Sabah.

#### PhD research participations and achievements

1. Shell Inter-varsity Student Paper Contest 2008 (S-SPEC), 3-4 March 2008, Universiti Teknologi Malaysia, Skudai – *1st Runner Up in Postgraduate Oral Presentation*.
2. Engineering Design Exhibition (EDX 21), 16-17th April 2008, UTP – *Gold for Chemical Engineering Postgraduate, Winner of the Best Postgraduate Presenter, 1st Runner Up for the Best Postgraduate Poster and 2nd Runner Up for the Most Innovative Award*.
3. 19<sup>th</sup> International Invention Innovative and Technology Exhibition (ITEX 2008), Kuala Lumpur Convention Centre, 9-11 May 2008.
4. The invention and New Product Exposition (INPEX), 11-14 June 2008, Pittsburgh, USA – *Two Gold Medals in Manufacturing and Chemicals categories*.
5. BASF International summer course, Ludwigshafen, Germany, 21 July – 2 August 2008.
6. Malaysia Technology Exhibition (MTE 2009), 19-21 February 2009, PWTC KL, *Silver medal*.