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Characterization of nanocarbon particles using nitrogen adsorption analysis: Isotherm, pore type, pore size and BET surface area

S. Zainal Abidin[1,*], I.S. Mohamad[1,2], A.Y. Bani Hashim[3], N. Abdullah[4]

[1)] Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka,
Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[2)] Centre for Sustainability and Environment, Universiti Teknikal Malaysia Melaka,
Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[3)] Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka,
Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[4)] Department of Chemistry, Centre for Foundation Studies, Universiti Pertahanan Nasional Malaysia,
Kem Sungai Besi, 57000, Kuala Lumpur, Malaysia

*Corresponding e-mail: Wanieyza_2792@yahoo.com

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ABSTRACT – In this research, the characterization of the porous material which is nanocarbon was investigated using nitrogen adsorption technique. The porosity existence of nanocarbon is analysed by using BET method. Then, the surface area is measured from diameter and depth of the existed pores. The BET surface area obtained shows that MWCNT-OH has the largest surface area due to high distribution of micropores and existence of non-porous pore in the sample.

1. INTRODUCTION

Nanoparticles are a great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. The properties of materials change as their size approaches the nano-scale and as the percentage of atoms at the surface of a material becomes significant. Nanoparticles have a very high surface area to volume ratio. This provides a tremendous driving force for diffusion especially at elevated temperatures. The specific atomic structure of nanoparticle make it an interesting chemical and physical properties according to those of graphite and diamond [1]. Generally, the characterization techniques evaluations is done in order to yield important information in studying the effects of surface porosity and particle size for materials engineering. The characterization of the porous materials commonly used gas adsorption as a major technique [2] [3]. Compared to the other gases and vapours available in this earth, nitrogen gas has become a universally pre-eminent gas to be used because of its availability and could be used as adsorptive. Dewar and Ramsay [4] [5] are the one of the earliest researchers that studies the adsorption nitrogen and other gases at liquid air temperature approximately to 88 K by investigating the composition of the atmosphere and the separation of the noble gas. Then, the research regarding adsorption of nitrogen gas and other gases was conducted followed by other researcher but at a low-temperature studies by charcoal, including an extensive series of measurements over varies temperature by Homfray in 1910 [6]. Thus, this research aimed to study the behaviour of nanocarbon structure which using nitrogen adsorption techniques in order to yield essential information in studying the properties of surface porosity and particle size for materials engineering.

2. METHODOLOGY

For this research, Autosorb 6-B surface area and pore size analyser machine manufactured by Quantachrome Instruments were used. The analysis for this characterization testing were made by measuring the volume of nitrogen gas adsorbed at the specific pressures. The characterization testing were performed on these selected nanocarbon which are Multiwalled Carbon Nanotube (MWCNT-OH), functionalized MWCNT and Commercial Carbon Nanofiber (CNF). Table 1 shows the properties of the three nanocarbon particles.

Table 1 Properties of the CNT.

Properties	MWCNT-OH	Functionalized MWCNT	Commercial CNF
Purity (%)	90	95	98
Outer Diameter	10-30nm	30-50nm	100nm
Specific Surface Area (m^2/g)	100	60	43
Form	Powder	Fine Powder	Powder

3. RESULTS AND DISCUSSION

The isotherm plot indicates the distribution of different type of pore which consists of micro, meso and macropores from an adsorption isotherm. The micropore diameter size is between 0 to 2nm, mesopore is between 2 to 50nm and macropore size is more than 50nm. According to Figure 1, MWCNT-OH isotherm can be classified as Type II and Type III. This isotherm type is produced by the non-porous or solid macro pore which showed non-porous solid characteristics. For functionalized MWCNT, the isotherm can be classified as Type II and Type IV. Commercial CNF can be classified as Type I and Type IV isotherm which means that the sample is mostly microporous with hysteresis loops.

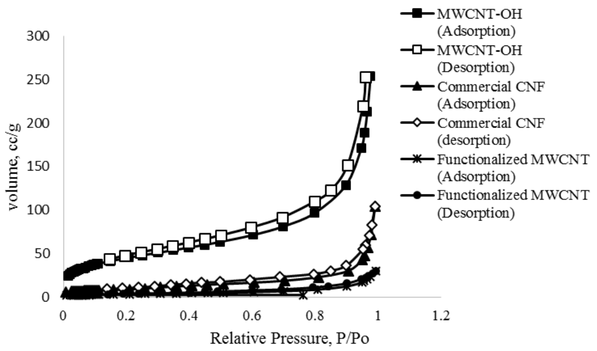

Figure 1 Isotherm comparison for MWCNT-OH, functionalized MWCNT and Commercial CNF.

Referring to the Figure 2, MWCNT-OH shows that the high peak is representing range of micropore type with diameter 1.4nm and depth 2.15×10^{-2} cc/g which shows that it has a high distribution of micropores and average distribution of mesopores. For functionalized MWCNT, the plot shows that high peak is representing range of mesopore type with diameter 3.1nm and depth 5.25×10^{-3} cc/g. Whilst, Commercial CNF shows the high peak with high distribution of micropores which have 0.39nm and depth 2.07×10^{-4} cc/g and moderate distribution of mesopores. Materials with micro-sized pores shows the strong interactions among the narrow pore walls with material adsorbed on the relative pressure which cause the volume of material adsorbed in the pores increases. Pore with meso-sized shows an increase in the volume of the adsorption due to the effect of the occurrence of capillary condensation turn through the establishment of a hysteresis loop. Macro-sized pores indicate the formation of a monolayer on relatively at low pressure and the adsorption layers at relatively high pressure. Material which have bigger pore will result in a weak interaction between materials adsorbed and the adsorbent which causing difficult adsorption.

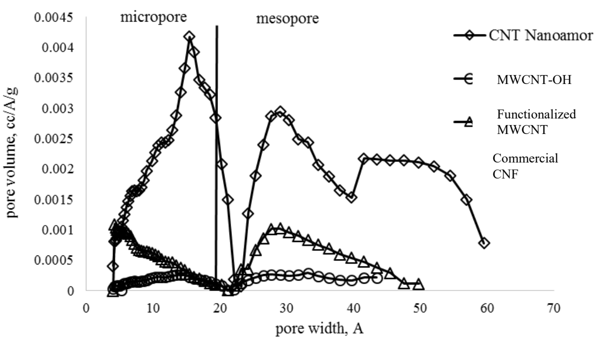

Figure 2 DFT pore size distribution comparison.

Surface area is measured from diameter and depth of existed pores whether it is coming from micro, meso or macro types influencing on surface area reading based on volume of gas adsorbed. Table 2 shows the BET surface area for all sample tested.

Table 2 BET surface area.

Nanocarbon	BET Surface Area (m^2/s)
MWCNT-OH	1.610×10^2
Functionalized MWCNT	1.261×10^1
Commercial CNF	3.241×10^1

The BET surface area obtained shows that MWCNT-OH has the largest surface area (MWCNT-OH > Commercial CNF > Functionalized MWCNT). This is due to the high distribution of micropores in the sample which contribute to the high surface area. The functionalized MWCNT has the smallest surface area due to the high distribution of mesopores in the sample.

4. CONCLUSION

Characterization using nitrogen adsorption analysis reveal the pore type of the material tested. From the result, MWCNT-OH nanocarbon has a higher BET surface area which is 1.610×10^2 m^2/s due to the non-porous or solid macro pore. Whilst, Functionalized MWCNT has the smallest BET surface area (1.261×10^1 m^2/s) due to the high distribution of mesopores in the nanocarbon. For Commercial CNF, the sample shows the microporous pore type with BET surface area 3.241×10^1 m^2/s. In conclusion, the BET characterization technique encompasses external area and pore area evaluations to determine the total specific surface area in order to yield important information in studying the effects of surface porosity and particle size for materials engineering.

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