CHAPTER IV
RESULTS AND DISCUSSIONS

4.1 Carbon Nanotubes Characterization

In order to study nanomaterials, it is vital to perform characterization studies to understand the nature of these materials. Ever since the discovery of carbon nanotubes, there has been much emphasis on their production and characterization because of their potential uses as strength and electronic materials, sensors, and gas storage. There are a variety of processes used to produce carbon nanotubes. They include the arc-discharge, laser ablation, and chemical vapor deposition. Hence, the carbon nanotubes produced from these processes need to be characterized to answer questions related to purity, quality, and type of carbon nanotubes being produced. We have used the following techniques to characterize the potential gas storage material: 1) Transmission Electron Microscopy, 2) Raman Spectroscopy, 3) BET Measurements and 4) X-ray diffraction

The results showed that as-received MWNTs were high purity open-ended multi-wall carbon nanotubes as shown in Figure 4.1. and Figure 4.2.. According to Raman Spectroscopy, shown in Figure 4.3 and Figure 4.4, there was no signal of SWNTs in as-received MWNTs. BET surface area of MWNTs is almost fourfold compared to commercial activated carbon. Figure 4.5 shows that 2-theta of as-received MWNTs is 25.720, compared with of graphite and carbon nanotubes in literature, Wu *et al.* (2000), which are 26.4 and 25.6, respectively.

4.1.1 Transmission Electron Microscopy

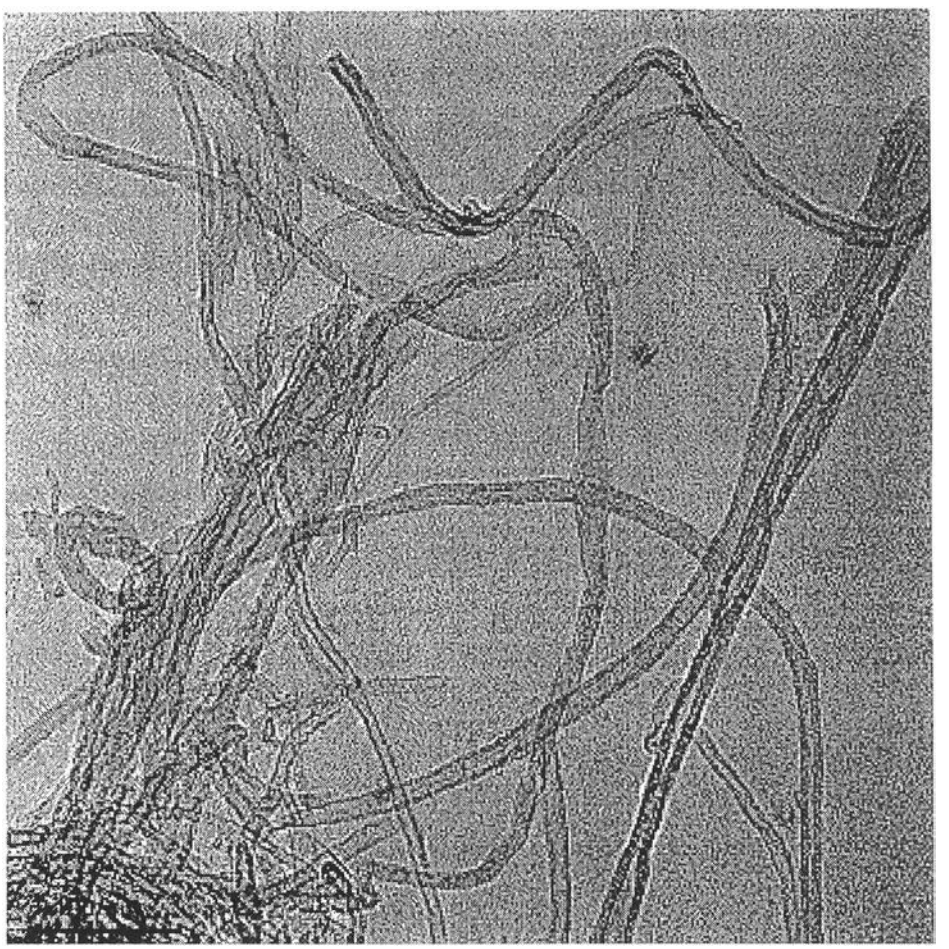

Figure 4.1. TEM image of as-received MWNTs showing the bundles of MWNTs, which are clearly visible, the 10 nm scale is shown in the bottom left corner.

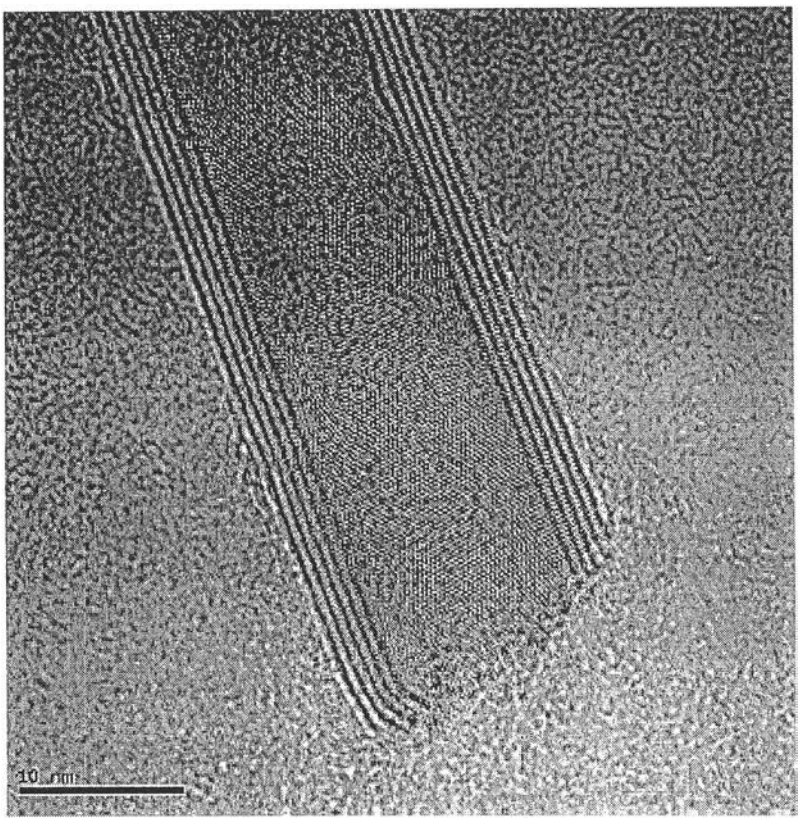

Figure 4.2. TEM image of as-received MWNTs showing clearly open-ended of MWNTs and their shells, the 10 nm scale is shown in the bottom left corner.

4.1.2 Raman Spectroscopy

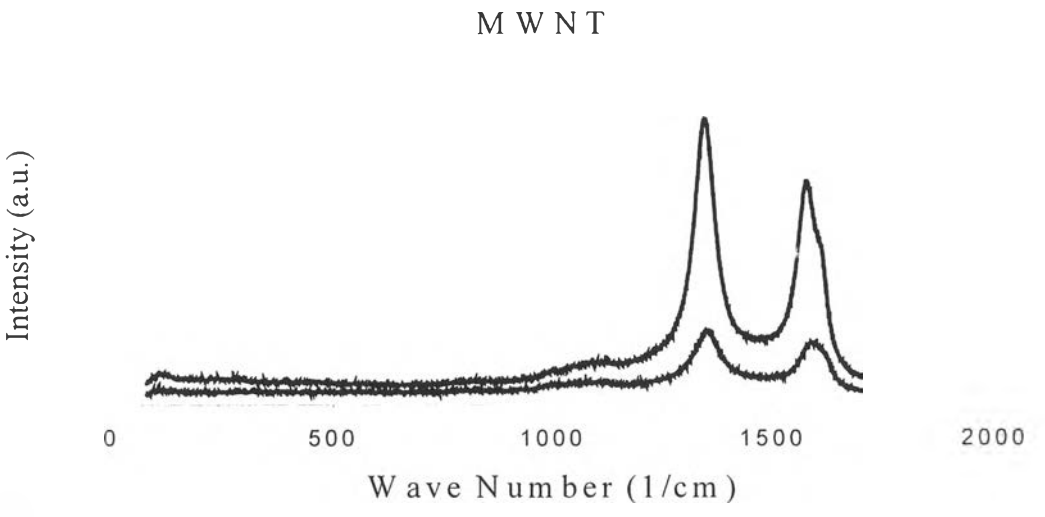

Figure 4.3. Raman spectroscopy of multi-walled carbon nanotube

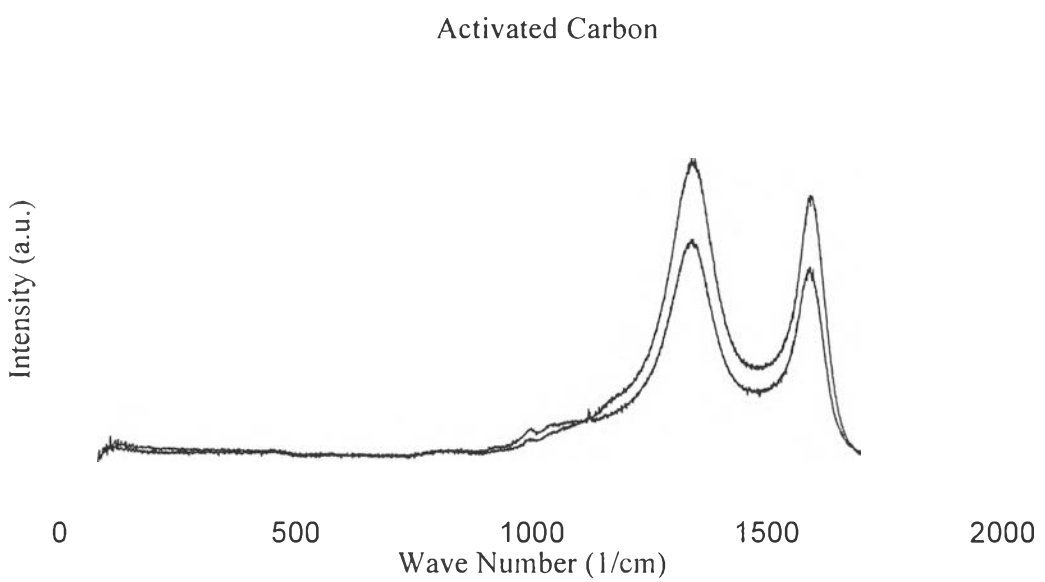

Figure 4.4. Raman spectroscopy of activated carbon

4.1.3 BET Surface Area

BET surface area of MWNTs and AC is 1063 m^2/g and 290 m^2/g, respectively.

4.1.4 X-ray Diffraction

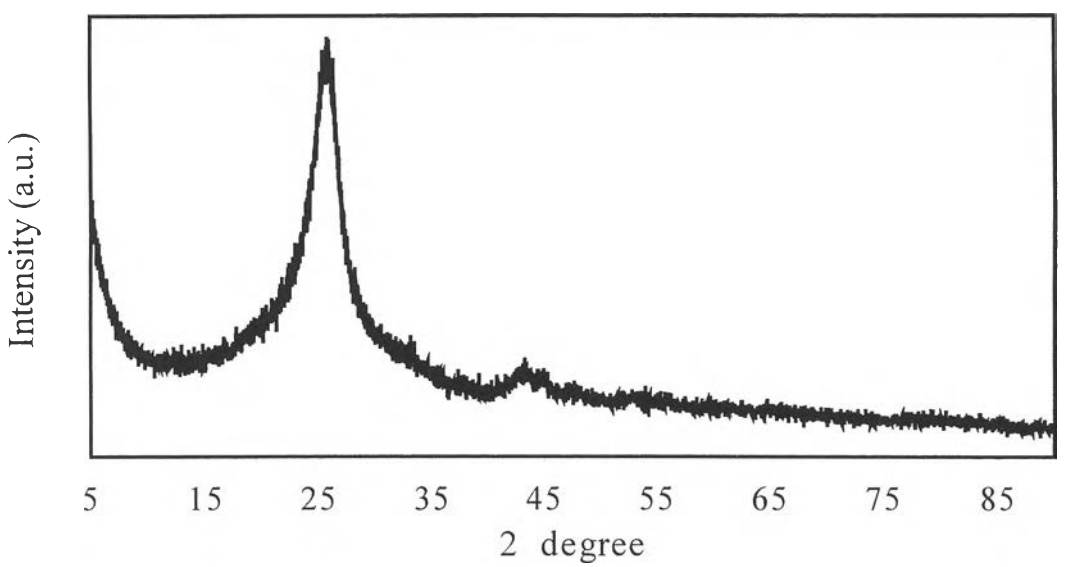

Figure 4.5. XRD of multi-walled carbon nanotubes

4.2 Calibration of Volume Spaces

We carried 8 runs to collect data for without adding known volume, varying from 136-142 psia and 4 runs for with adding known volume (20.397 cc) at ~141 psia. A steady-pressure time was 10 minutes. The average pressures of raw data for 10 minutes before and after helium expansion time were used to represent pressure values in each run. Standard deviation of raw data was not greater than 0.2 % conforming to global error of pressure transducer.

The volumes of manifold and sample cylinder were calculated by matching each value from 2 systems, with/without adding known volume. The result showed that the calculated volume of manifold were in range of 164-170 cc and in range of 52-54 cc for the calculated volume of sample cylinder.

In conclusion, the calculated average volume of manifold and sample cylinder used to represent in this thesis is 166.53 cc and 53.43 cc, respectively.

4.3 Collecting of Adsorption Data

The average pressures of raw data for 10 minutes after each calculated equilibrium time were used to represent pressure value. Standard deviation of raw data is not greater than 0.2 % conforming to global error of pressure transducer.

4.4.1 The Effect of Calculated Equilibrium Time

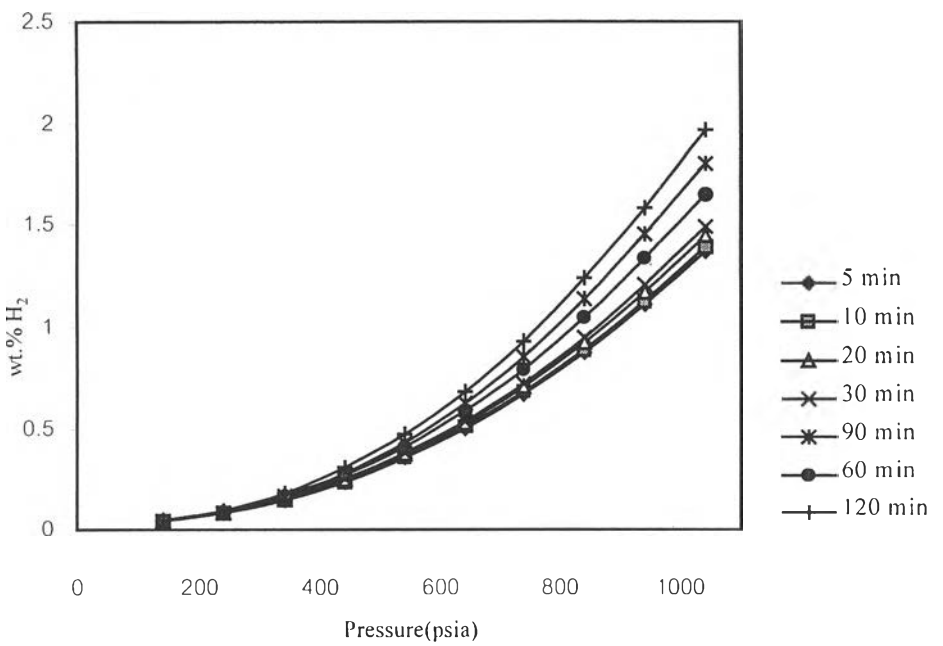

Figure 4.6. wt% H_2 of 7 g MWNT, method 2 at varying calculated equilibrium time

Figure 4.6. shows the plot between wt% hydrogen and equilibrium pressure at different calculated equilibrium time of multi-walled nanotubes 7 g, method 2. Results are varying from less than 0.1 to ~ 2 wt.%H_2. The higher pressure, the higher wt.%H_2 adsorbed. The more calculated equilibrium time, the higher deviate in wt.%H_2 at high pressure. There are two possible reasons. One is the system did not reach the equilibrium, yet. Another is there is probably feasible leak of gas in the system.

4.4.2 The Effect of Studying Method

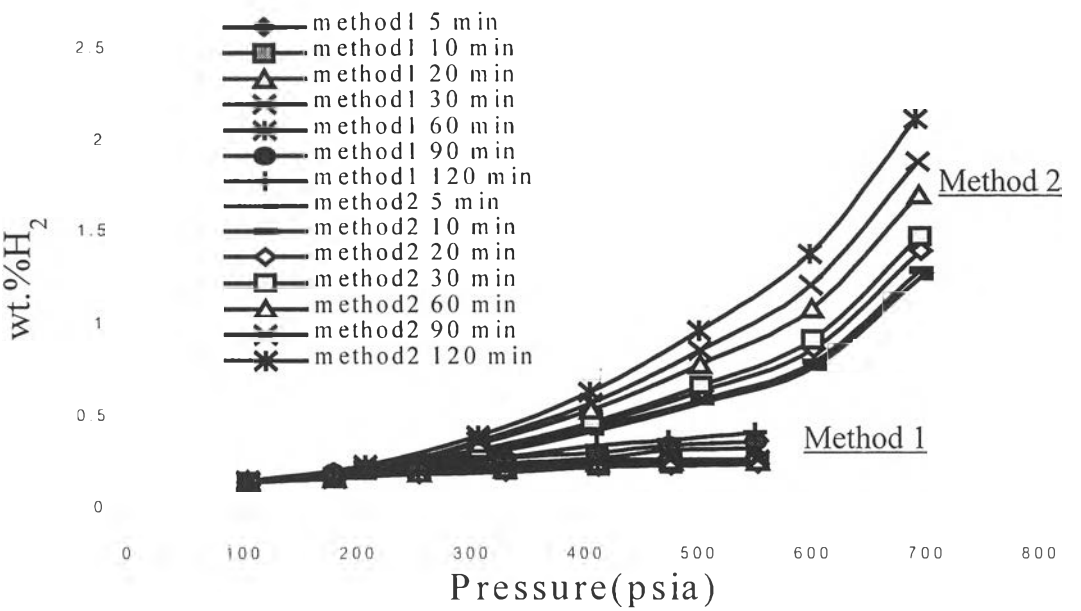

Figure 4.7. wt% H_2 of 2 g MWNT at varying calculated equilibrium time and different method

Figure 4.7. shows the plot between wt% hydrogen and equilibrium pressure at different calculated equilibrium time of multi-wall nanotube 2 g, compared between method 1 and 2. Results are on the same trend. The higher pressure, the higher wt.%H_2 adsorbed. The more calculated equilibrium time, the higher deviate in wt.%H_2 at high pressure. Furthermore, the deviation of obtained result of method 1 is much less than method 2 especially at high pressure. Method 2 gives higher value hydrogen adsorbed.

4.4.3 The Effect of Amount of Adsorbent

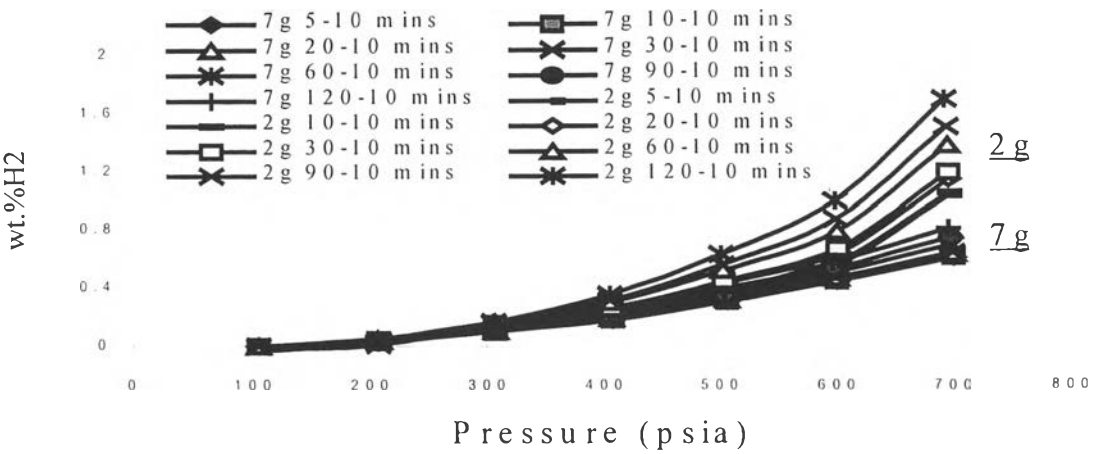

Figure 4.8. wt% H_2 of MWNTs method 2 at varying calculated equilibrium time and amount of adsorbent

Figure 4.8. shows the plot between wt% hydrogen and equilibrium pressure at different amount of multi-wall carbon nanotube, method 2. Results are on the same trend. The higher pressure, the higher wt.%H_2 adsorbed. The more calculated equilibrium time, the higher deviate in wt.%H_2 at high pressure. Furthermore, the deviation of obtained results of 7 g is much less than 2 g especially at high pressure. 2 g MWNTs gives higher value hydrogen adsorbed.

4.4.3 The Effect of Background Leak

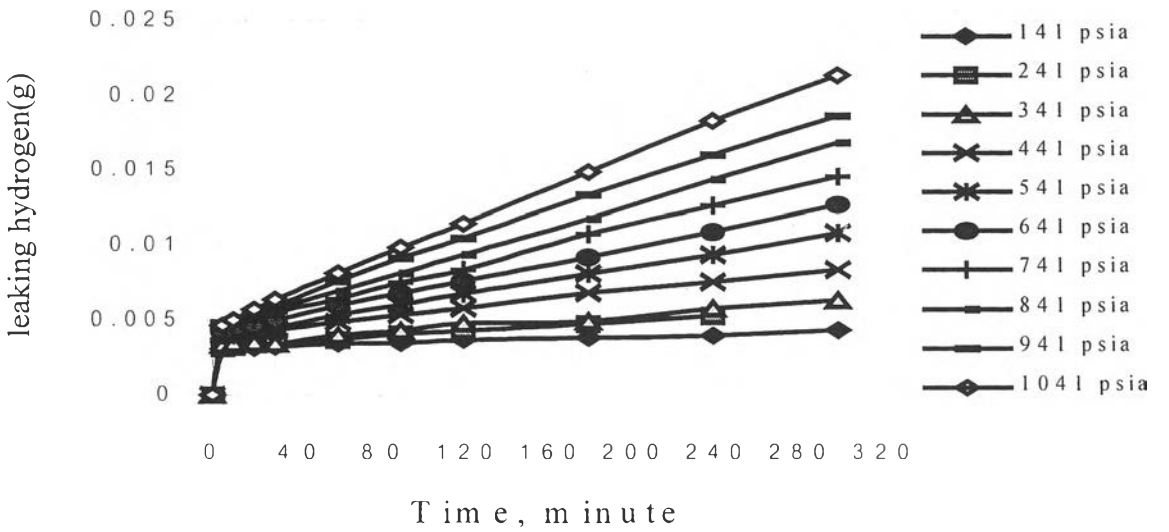

Figure 4.9. Amount of leaking hydrogen of blank of method 1 at varying calculated time

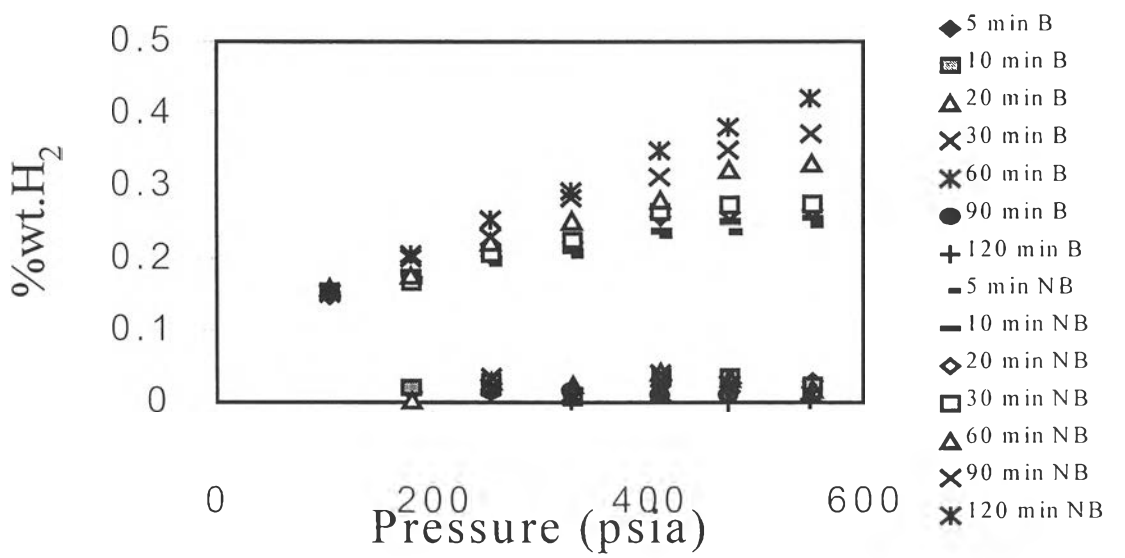

Figure 4.10. MWNT 2 g method 1compared between with/without leak test

(NB = Not subtract blank test, B = Subtract blank test)

Figure 4.9. shows the plot between leaking amount of hydrogen (gram) at various time of blank method 1. Results are on the same trend. The higher pressure, the more leaking amount. The more time, the more leaking amount. It expressed that the system is not leak-free, thus, leak background must take into account in reported result analysis.

Figure 4.10. shows the plot between MWNT 2g, method1, compared between subtract blank test and without subtract blank test. The results showed that after taking blank test into account, the results were decreased dramatically. 2g MWNT adsorbed almost nothing. The result that did not take blank test into account was not reliable.

4.4.4 The Effect of Type of Adsorbent

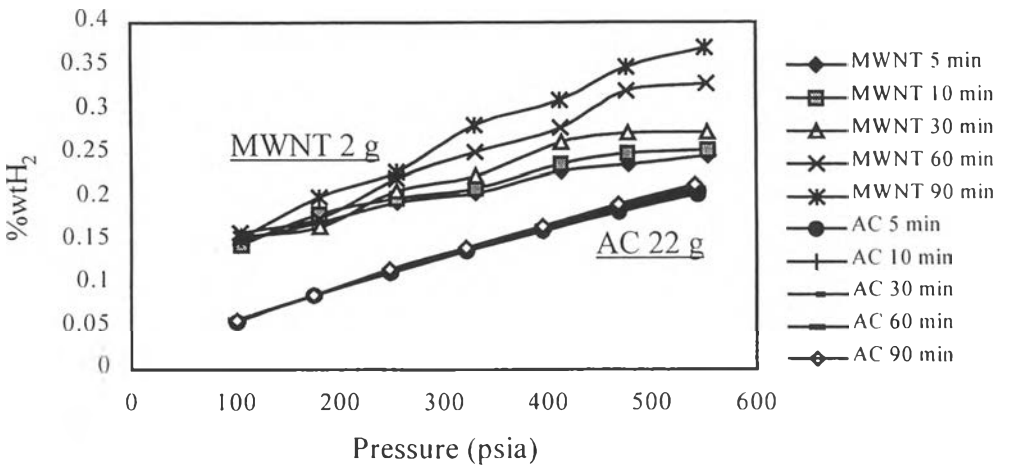

Figure 4.11. Method 1-compared between MWNT 2g and AC 22g without take effect of background leak into account

Figure 4.11. shows that $\%wtH_2$ of MWNT is higher than AC at any pressure, but the result express the same trend for MWNT that higher pressure and higher calculated time obtained higher deviation in the result. In contrast, the deviation AC sorption value results are close to each others at any calculated time and pressure even both of AC and MWNT do not take effect of background leak into account. It is strongly implied that with sufficient amount of adsorbent the result is more reliable and it could compensate any feasible errors of the experiment.