CHAPTER IV
RESULTS AND DISCUSSION

This study used the Single Walled Carbon Nanotubes (SWNTs) that was produced by CoMoCAT® process and silica gel was used as the support catalyst. The SWNTs sample contained SWNTs about 1 to 25wt%, metal about 1 to 6wt% and the silica gel which more than 70wt%. Therefore, the more removal of silica gel, the higher purity of SWNTs is obtained (Chungchamroenkit, 2004). In this research, both concerned the sodium hydroxide (NaOH) and triethylenetetramine (TETA) were used to remove silica. The effects of amount of either NaOH or TETA, temperature and reaction time were studied. The use of sonication to enhance the silica dissolution was also investigated. The optimum conditions for the maximum percentage of silica dissolved were applied to the froth flotation experiments purify the single walled carbon nanotubes (SWNTs). The combination of Laser Raman Spectroscopy and Temperature Programmed Oxidation (TPO) techniques were used to characterize both qualitative and quantitative of purified SWNTs. The Laser Raman Spectroscopy was used for systematic screening of the all types of carbon products and the Temperature Programmed Oxidation (TPO) was used to confirm the forms of carbons presenting in samples and also to provide the amount of total carbon products.

4.1 Silica Dissolution Results

4.1.1 The Effect of Triethylenetetramine Volume

An amount of 1.5 g of silica was dissolved in different volumes of triethylenetetramine at 70°C for 1 hour. Table 4.1 and Figure 4.1 summarize the result of percentage of silica dissolved at different amount of TETA. When the TETA volume was increased the silica dissolution increased until at the 250 mL of TETA gave the silica dissolved reached the maximum value at about 73%. Therefore, a TETA volume of 300 mL was used for further experiments to ensure that the studied conditions were not limited by TETA.

Table 4.1 The amount of silica dissolved at different volumes of TETA, 70°C and 1 hour

TETA (mL)	% Silica Dissolved
50	16.22
100	32.50
150	50.99
250	72.90
300	73.18
400	74.40

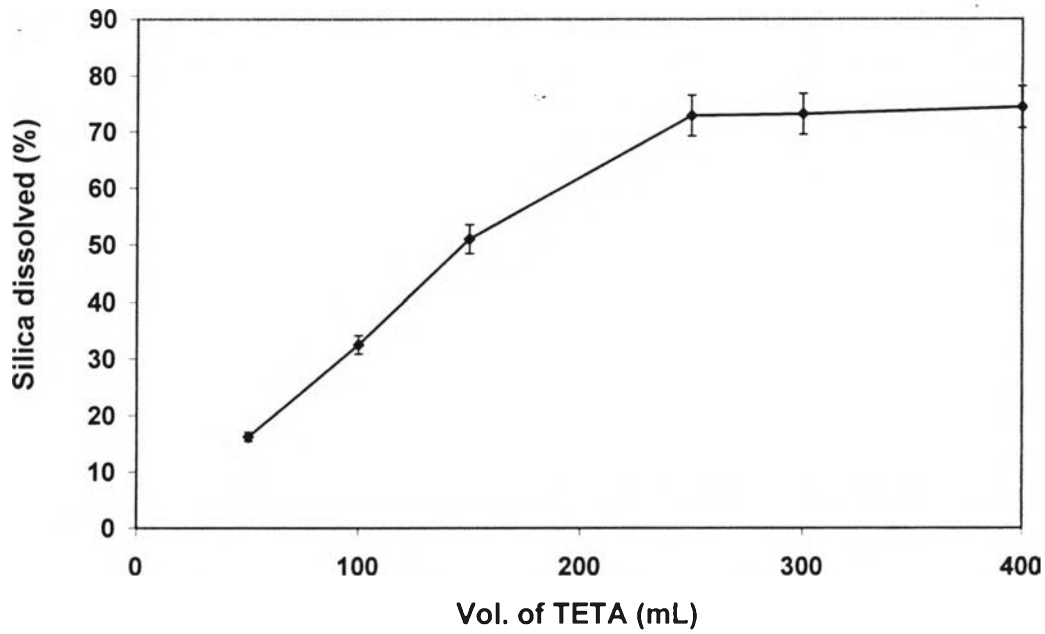

Figure 4.1 Effect of TETA volume on silica dissolved at 70°C for 1h.

4.1.2 The Effect of Temperature

1.5 g of silica and 300 mL of TETA were used in this study. The dissolution temperature was varied whole the dissolution time was kept constant at 1 hour. The results are shown in Table 4.2 and Figure 4.2. When the temperature increased, the silica dissolved also increased and it reached the maximum value of 73%. After that with increasing the temperature, the silica dissolved decreased remarkably. A drastic decrease in the percentage of silica dissolved with increasing temperature especially higher than 70°C results from the decomposition of TETA. Hence, the optimum temperature is 70°C was used in next experiments.

Table 4.2 The amount of silica dissolved at different temperatures (TETA volume = 300 mL and dissolution time = 1 hour)

TETA (mL)	% Silica Dissolved	Temp (°C)
300	35.64	26 (RT)
300	53.77	50
300	65.03	60
300	73.18	70
300	50.21	100
300	41.14	140

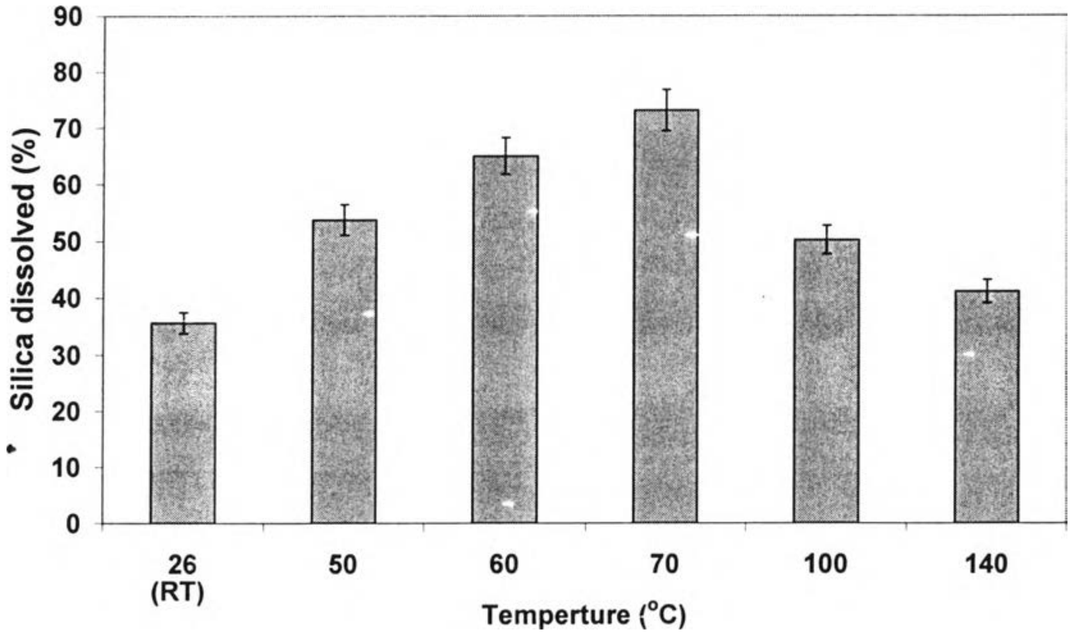

Figure 4.2 Effect of temperature on the silica dissolved at TETA volume 300 mL for 1h.

4.1.3 The Effect of Reaction Time

1.5 g of silica and 300 mL of TETA were used in this study. The temperature was kept constant at 70°C while the reaction time was varied. The results are shown in Table 4.3 and Figure 4.3. When the reaction time is increased the silica dissolved increase until at 3 hours which gave the silica dissolved at about 73%. After that, increasing the reaction time make the dissolved silica decreased. This effect may be TETA polymerized or be decomposed at the longer reaction time. So, the optimum reaction time is 3 hours.

Table 4.3 The amount of silica dissolved at different reaction time

TETA (mL)	% Silica Dissolved	Time (h)
300	73.18	1
300	77.40	3
300	73.02	5
300	72.74	10

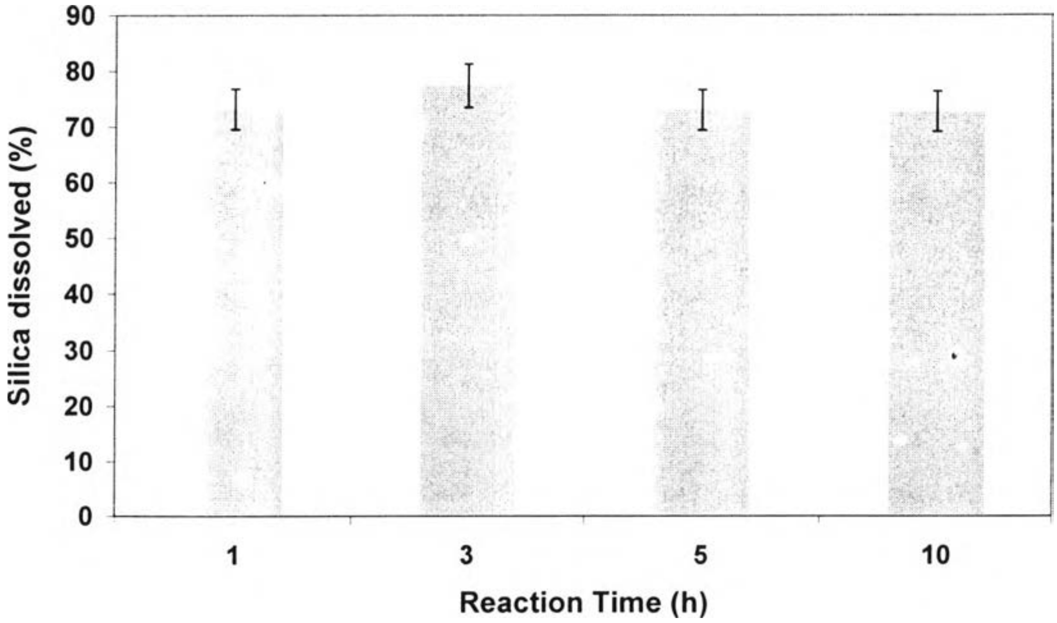

Figure 4.3 Effect of reaction time on the silica dissolution at TETA volume 300 mL at 70°C.

4.1.4 The Effect of Sodium Hydroxide Concentration

In this study 1.5 g of silica was dissolved in 50 mL of sodium hydroxide (NaOH). The NaOH concentration was varied while the temperature and the reaction time were kept constant at 70°C and 1 hour, respectively. Table 4.4 and Figure 4.4 summarize the results of percentage of silica dissolved at different NaOH concentration. When the NaOH concentration increased, the silica dissolved increased almost linearly and reached 100% silica dissolved at 1 M NaOH.

Table 4.4 The amount of silica dissolved at different NaOH concentration at 70°C for 1 hour

NaOH (M)	% Silica Dissolved
0.25	26.65
0.50	53.29
0.75	80.15
1.0	99.78
3.0	99.82
5.0	99.90

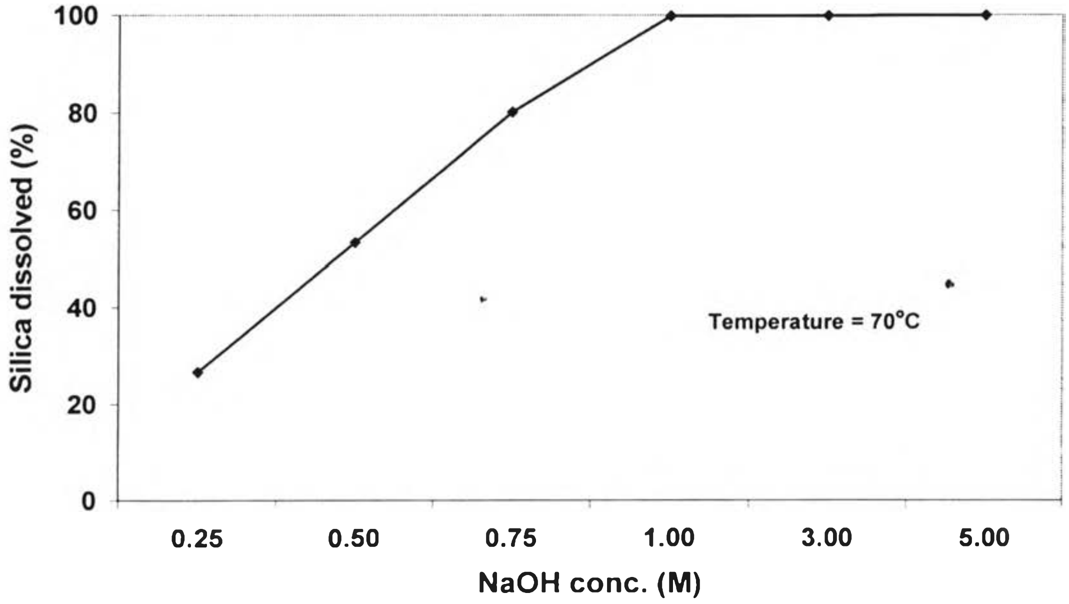

Figure 4.4 Effect of NaOH concentration on the silica dissolution at 70°C for 1 hour.

4.1.5 The Effect of Temperature

1.5 g of silica was dissolved in 50 mL sodium hydroxide solution containing different concentrations. The temperature was kept constant at room temperature and 70°C while the reaction time was set 1 hour. The results are shown in Table 4.5 and Figure 4.5. From any given NaOH concentration, an increase in temperature affected the silica dissolution insignificantly.

Table 4.5 The amount of silica dissolved at two different temperatures

NaOH (M)	% Silica Dissolved	Temp (°C)
0.25	23.99	RT*
	26.65	70
0.50	50.41	RT
	53.29	70
0.75	78.66	RT
	80.15	70
1.00	99.02	RT
	99.78	70
3.00	99.79	RT
	99.82	70
5.00	99.89	RT
	99.90	70

* Room temperature = 26 °C

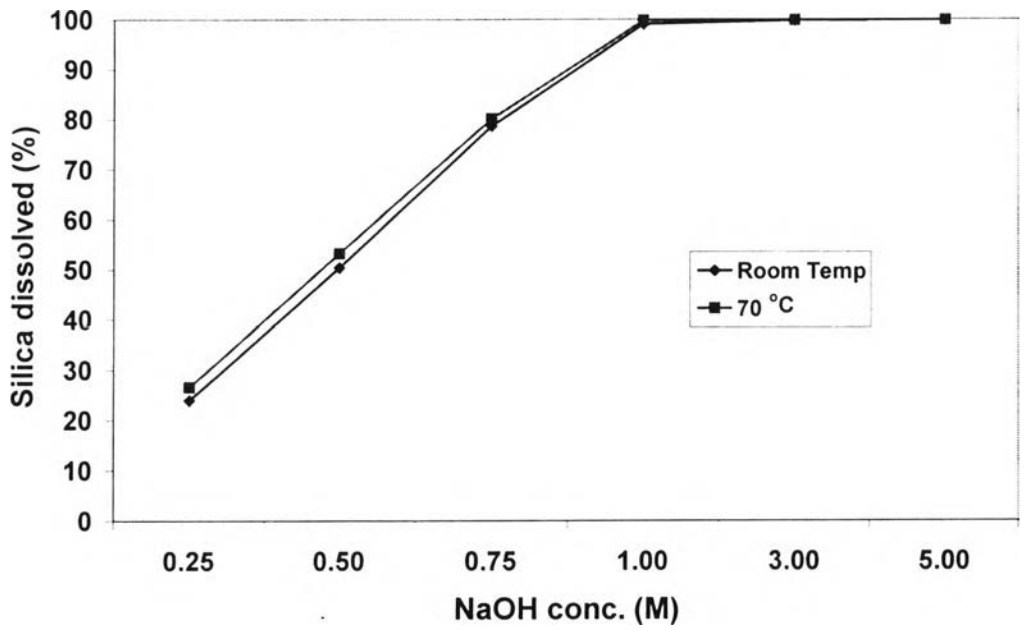

Figure 4.5 Effects of temperature and NaOH concentration on silica dissolution at 1 hour reaction time.

4.2 Sonication and Froth Flotation Results

In this experiment, the AP-SWNTs produced by the CoMoCAT® process which silica was used as catalyst support contained 1 to 25 wt% carbons, 1 to 6 wt% metals and 69 – 98 wt% silica. For the step of silica dissolution by either NaOH or TETA, sonication was used to enhance the dissolution reaction. The Froth flotation was further used to concentrate the purify SWNTs after the sonication. For the carbon nanotubes characterization, Laser Raman Spectroscopy and Temperature Programmed Oxidation (TPO) techniques were employed to quality and quantity both of the samples before and after the froth flotation step. It can provide information about purity, distribution of tube diameters, and electronic properties.

The Raman Spectrum results of carbon nanotubes give three important modes, namely graphite-like tangential mode (G-band), disordered carbon band (D-band), and the radial A_{1g} breathing mode frequency range (RBM). The tangential modes or G-band locates at around 1570 cm^{-1}, which are the characteristics of both carbon nanotubes and graphite. The latter band at 1350 cm^{-1} (D-band) corresponds to the disorder or amorphous carbon. The presence of the RBM, which locates at frequencies below 300 cm^{-1}, can provide information about the existence of SWNTs in the sample and it can be used to estimate the diameter of SWNTs. The intensity of D-band relative to that of G-band can be used as a qualitative measurement for the formation of undesired forms of carbon. Moreover, the D-band to G-band (D/G) intensity ratio also provides a relative amount of disorder carbon to graphite-like carbon. Typically, if the relative intensity ratio (D/G) is low, this can indicate that the formation of disorder carbon is lower than graphite-like carbon, so it means that carbon mostly consists of graphite-like forms such as SWNTs, MWNTs, and nanofiber. On the contrast, if the relative intensity ratio (D/G) is high, this can imply that the sample is mainly in disorder carbon form.

The results of Temperature Programmed Analysis were also used in this work for quantifying the amount of total carbon products. All of the carbonaceous products are oxidized to generate carbon monoxide and/or carbon dioxide. The amount of the total emitting gas gives a direct measurement of the amount of all carbon products. This method is usually called Temperature Programmed Oxidation (TPO). Temperature Programmed Oxidation (TPO) was used to confirm the types of carbons in all samples. Carbons with different morphology are oxidized at different temperatures leaving to identifying types of carbon products. In 2000, Kitiyanan and coworkers investigated oxidation temperature for each type of carbon deposits. The oxidation temperature of amorphous carbon was found in the range of 300-400°C, while SWNTs and MWNTs were oxidized around 495-530°C and 600-700°C, respectively.

The sample TPO profile of the carbon produced on the Co:Mo/SiO_2 catalyst is illustrated in Figure 4.6. This sample had a small oxidation peak centered at around 330°C, which is assigned to amorphous carbon, and a major peak marked in the figure with an arrow, centered at about 510°C, which is ascribed as SWNTs. The TPO profiles of reference samples are shown comparatively in Figure 4.6. For the first reference, graphite powder was physically mixed with the Co:Mo catalyst. As shown in Figure 4.6, the oxidation of this form of carbon occurs at very high temperatures, starting at about 700°C. The second reference sample investigated was a commercial sample of purified SWNTs obtained from Tubes@Rice (Rice

University). The TPO of this sample exhibited two peaks, a low-temperature one that corresponds to the oxidation of the amorphous carbon and a second one centered at 510°C, which corresponds exactly to the position of SWNTs.

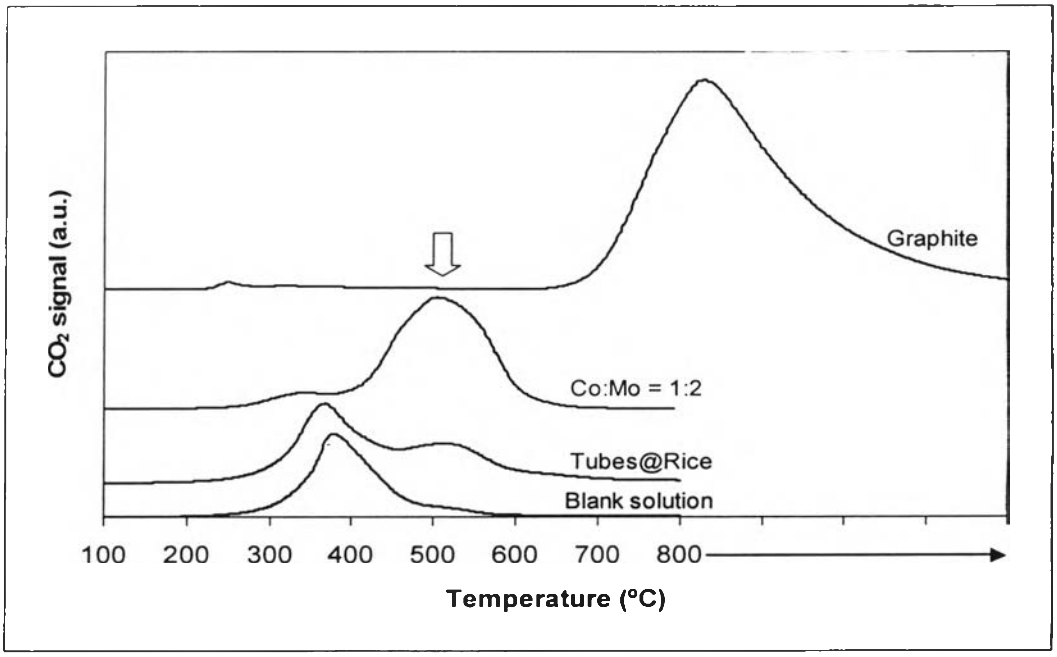

Figure 4.6 TPO profiles of all the carbonaceous species present the SWNTs sample produced on the Co-Mo 1:2/SiO_2 catalyst compared to those of graphite reference, blank solution, and the commercial SWNTs obtained from Tubes@Rice (Kitiyanan, 2000).

In order to study the effect of TETA volume, the as-prepared SWNTs were treated with different TETA volumes. The Laser Raman spectroscopy and TPO techniques were used to characterize the carbon samples. Figures 4.7 and 4.8 illustrate the Raman spectra and TPO profile of the sample produced from CoMoCAT® process. The Raman spectrum of carbonaceous product shows three main peaks of RBM, G-band, and D-band signals. The RBM signal corresponds to the presence of SWNTs, while the G-band and D-band represents graphite-like and disorder carbon, respectively. The intensity of D-band relative to G-band can be used as a qualitative measurement for the formation of undesired forms of carbon.

Moreover, the D-band to G-band (D/G) intensity ratio also provides a relative amount of disorder carbon to graphite-like carbon. Typically, if the relative intensity ratio (D/G) is very low, this indicates that the formation of disorder carbon is much lower than graphite-like carbon or it means that the carbon produced is mostly consisted of graphite-like form such as SWNTs, MWNTs, and nanofiber. On the contrary, if the relative intensity ratio (D/G) is very high, this implies that the sample is mainly in disorder carbon form. Figure 4.7 shows the Raman spectra of the as-prepared carbon nanotube sample. From Raman results, G-band and D-band as well as the RBM peak were found in the CoMoCAT® sample. As mentioned before, the RBM peak indicates the existence of SWNTs and the intensity ratio of D-band to G-band (D/G) provides a relative amount of disordered carbon to graphite-like carbon. From the Raman results, the relative intensity of D-band was significantly lower than of that of G-band indicating that the formation of amorphous carbon is much less than graphite-like carbon.

Figure 4.8 shows the TPO profile of the as-prepared SWNT sample from CoMoCAT® process. There were two major peaks of the higher peak centered at around 550°C corresponding to the oxidation of SWNTs and the smaller oxidation peak centered at around 450°C indicating the oxidation of amorphous carbon. According to both of the Raman and TPO results, most of products obtained are SWNTs. The carbon yield is estimated only 4 wt%.

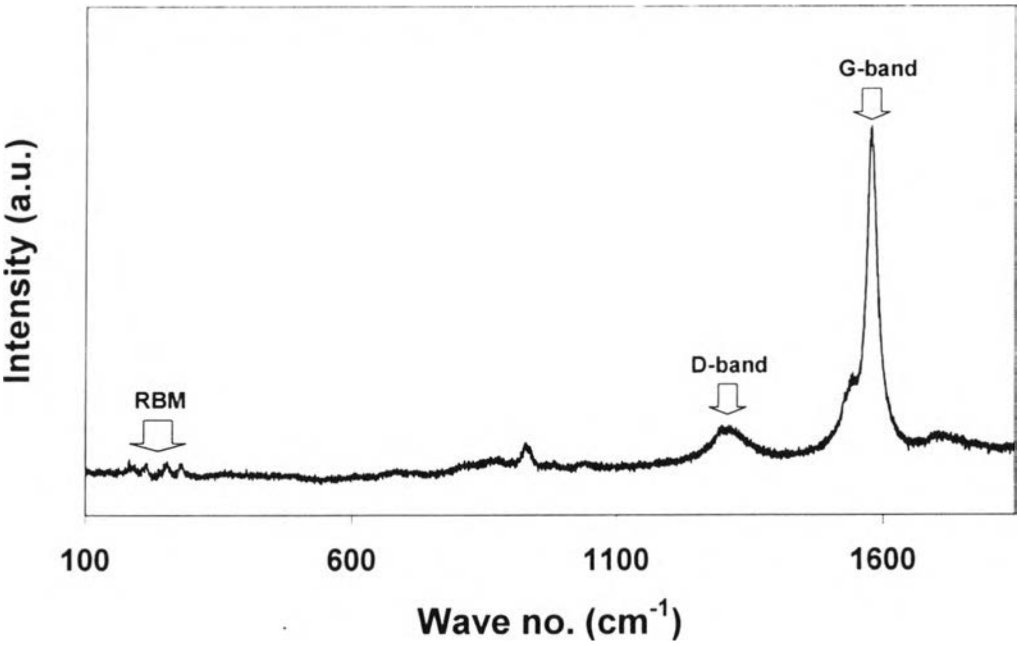

Figure 4.7 Raman spectra of as-prepared Carbon Nanotubs from CoMoCAT® process.

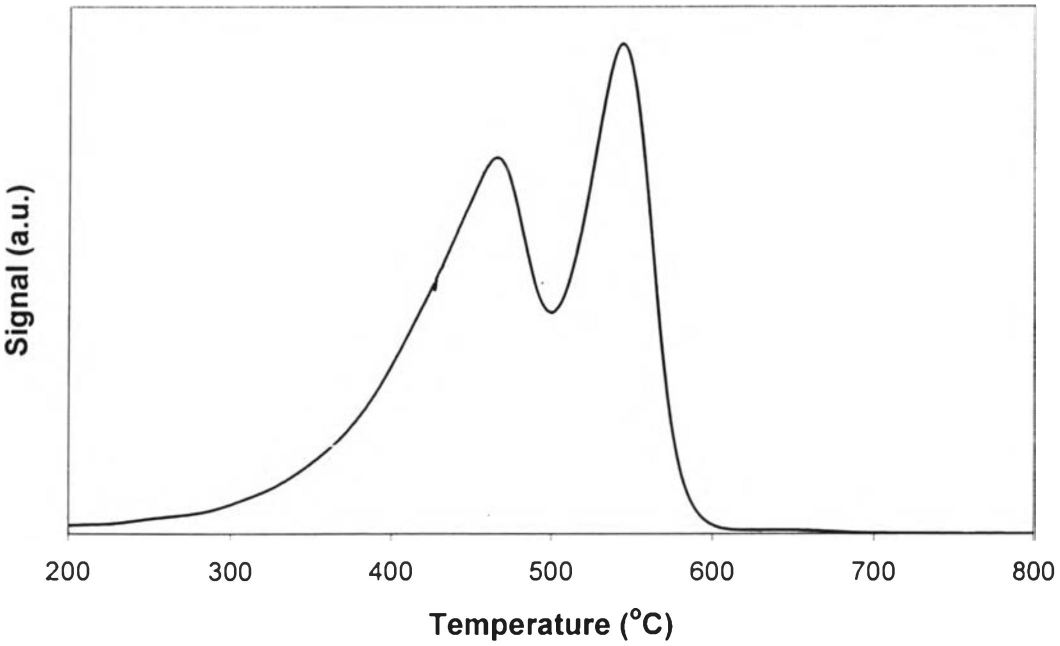

Figure 4.8 TPO profile of as-prepared carbon produced from CoMoCAT® process.

4.2.1 Sonication Effects

4.2.1.1 Sonication with TETA pretreatment

Table 4.6 summarizes the carbon purity improvement of as-prepared SWNT sample after TETA treatment with sonication by using the TPO technique. The Raman spectrum of as-prepared SWNT sample which was treated with distilled water for 3 hours of sonication at 70°C is shown in Figure 4.9. The graph shows that the D band was smaller than the G band indicating that the carbon in the sample is SWNTs more than amorphous forms. Moreover, the TPO profile of this sample is also confirmed as shown in Figure 4.10. The peaks occurred at 320 and 550°C, which correspond to the amorphous carbon and graphite carbon, respectively. The amount of carbon calculated from the area under peak of TPO profile was about 3.60 wt%. The result indicates that sonication can break partially the bond between the carbon nanotubes and the silica support.

Figures 4.11-4.14 show the effect of TETA volume and sonication time on carbon purity of SWNT sample. When the TETA volume increased, the percentage of carbon purity increased. As explained earlier, an increase in TETA quantity simply increase the dissolution of silica. From the results, the effect of sonication time was found to be insignificant indicating that the bond between SWNTs and the silica surface is very strong and cannot be broken by sonication.

Table 4.6 Purity of treated as-prepared SWNT sample at different TETA volumes, different sonications times and 70°C

Treatment Conditions	Carbon Purity (%)	RBM
50 mL Di-water, 3 h	3.60	yes
50 mL TETA, 30 min	22.00	yes
50 mL TETA, 1 h	23.42	yes
50 mL TETA, 3 h	23.72	yes
50 mL TETA , 7 h	22.79	yes
100 mL TETA, 30 min	23.93	yes
100 mL TETA, 1 h	24.74	yes
100 mL TETA , 3 h	25.88	yes
100 mL TETA , 7 h	24.75	yes
150 mL TETA , 30 min	27.60	yes
150 mL TETA , 1 h	29.92	yes
150 mL TETA , 3 h	31.52	yes
150 mL TETA, 7 h	30.57	yes
250 mL TETA , 30 min	30.55	yes
250 mL TETA, 1 h	31.94	yes
250 mL TETA, 3 h	32.91	yes
250 mL TETA , 7 h	32.41	yes

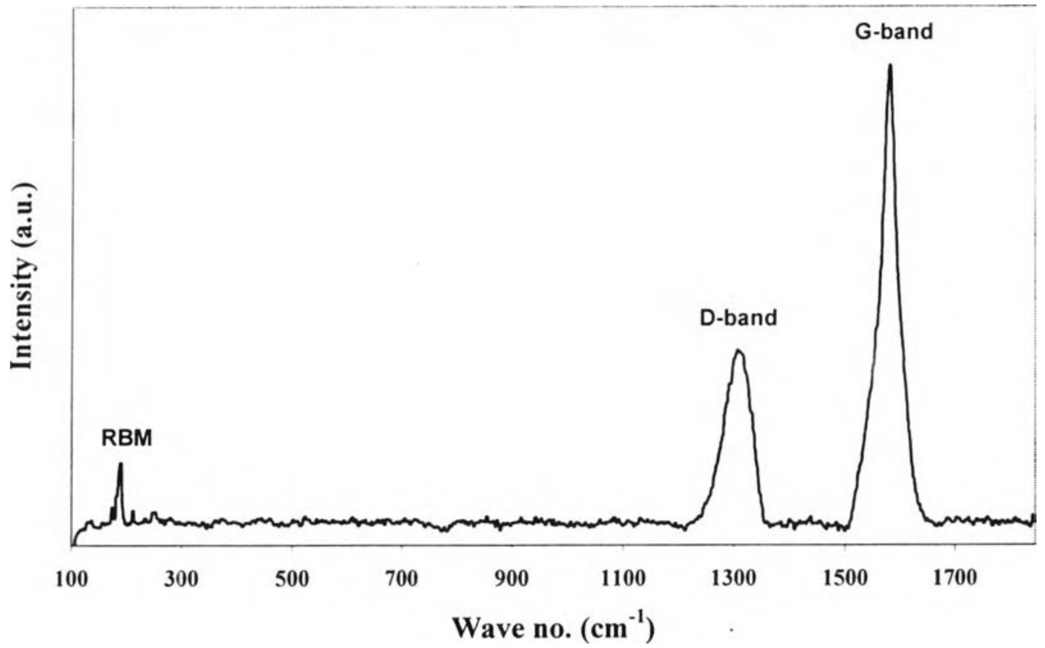

Figure 4.9 Raman spectra of as-prepared SWNT sample with distilled water at 70°C and for 3 hours sonication.

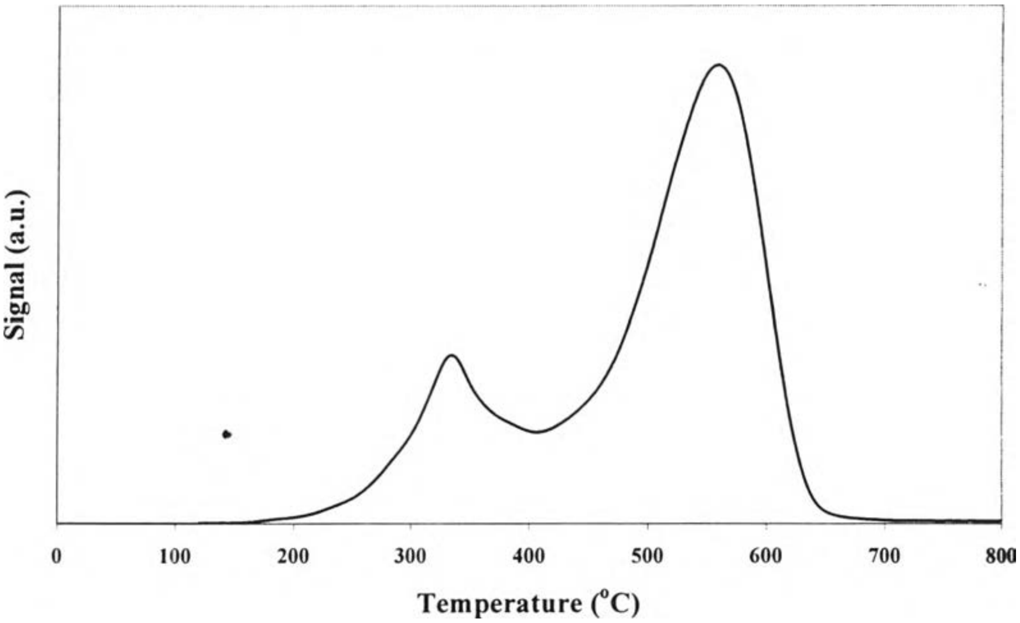

Figure 4.10 TPO profile of as-prepared SWNT sample treated with distilled water for 3 hours sonication at 70°C.

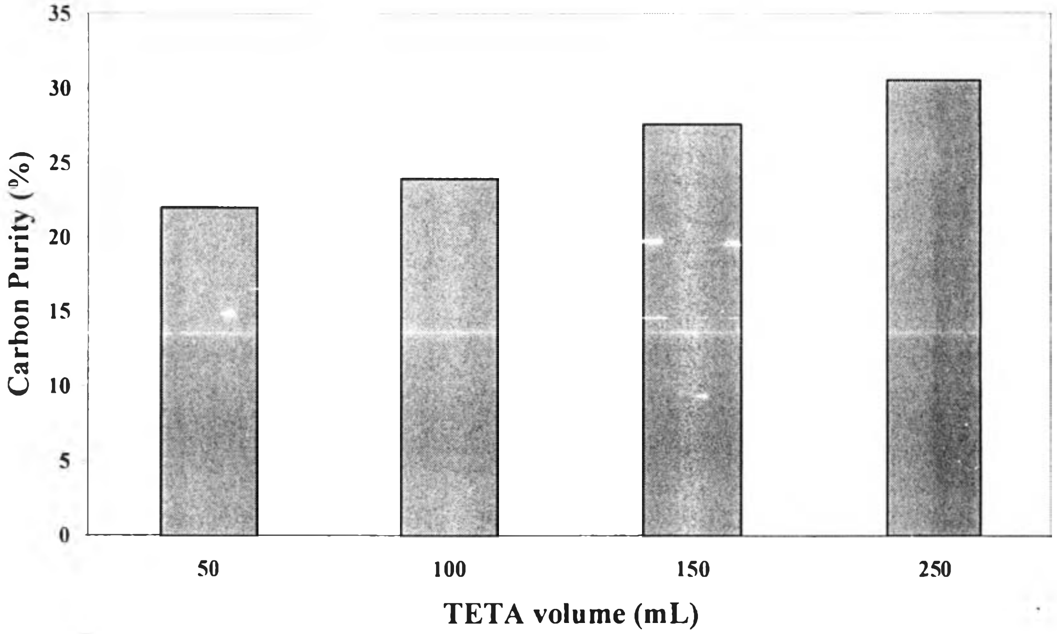

Figure 4.11 The effect of TETA volume on the percentage of carbon purity at 30 minutes of sonication and 70°C.

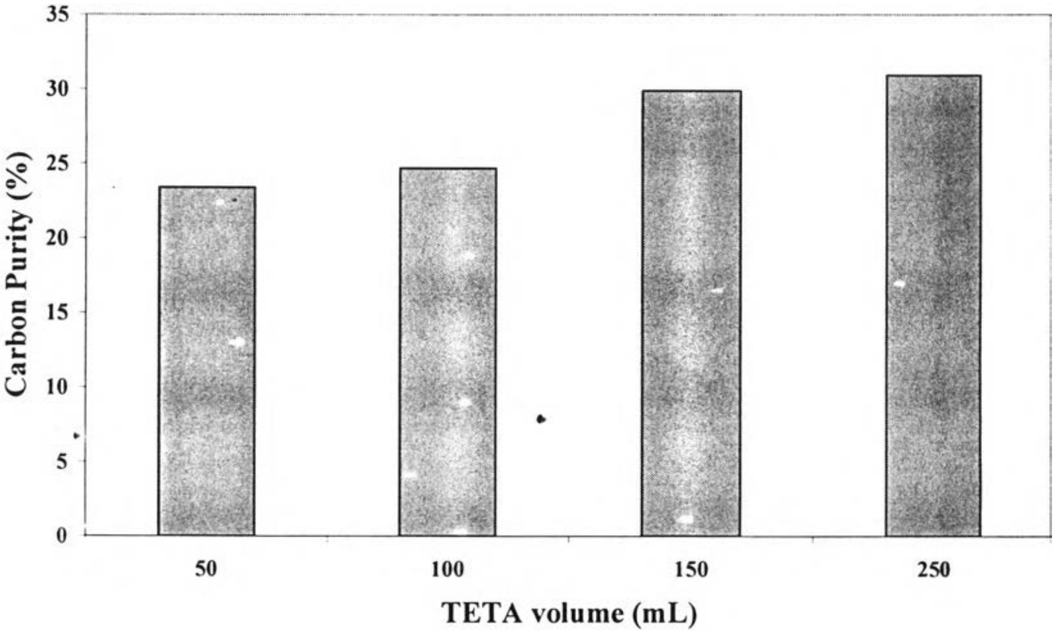

Figure 4.12 The effect of TETA volume on the percentage of carbon purity at 1 hour of sonication and 70°C.

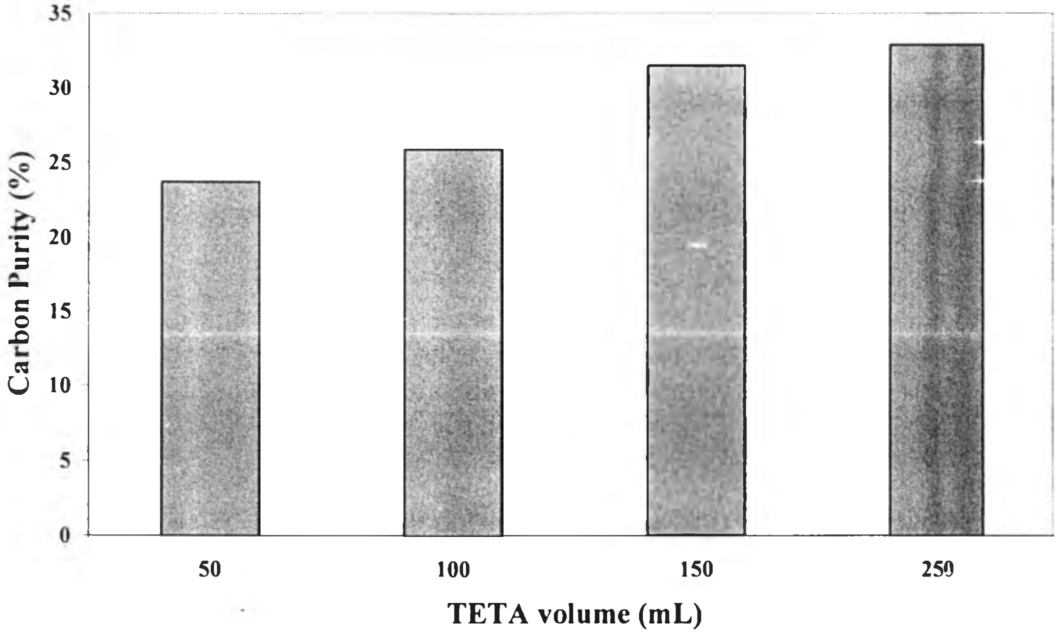

Figure 4.13 The effect of TETA volume on the percentage of carbon purity at 3 hours of sonication and 70°C.

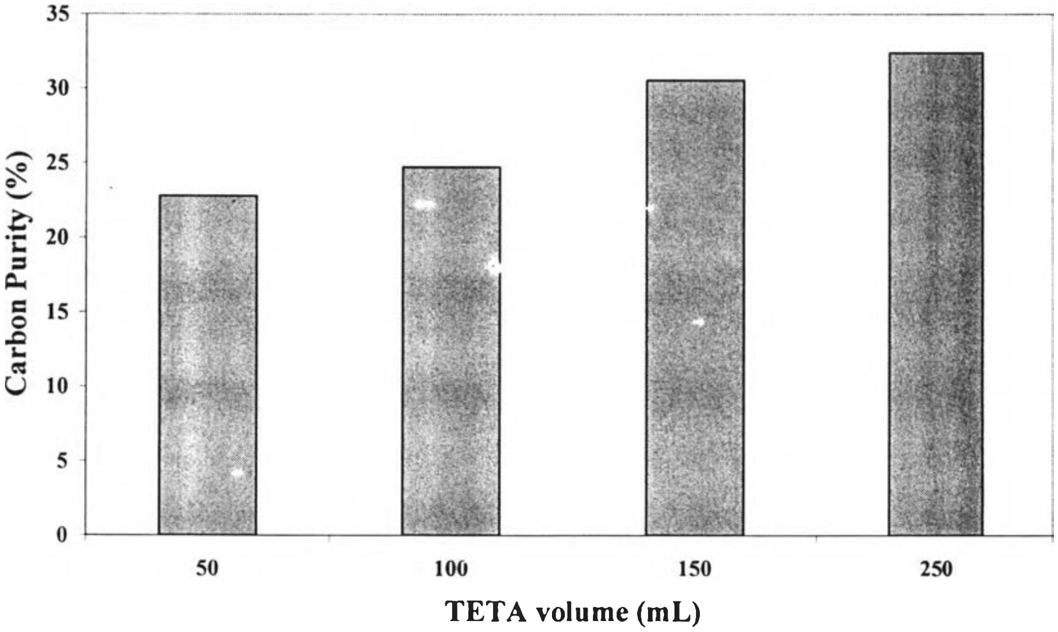

Figure 4.14 The effect of TETA volume on the percentage of carbon purity at 7 hours of sonication and 70°C.

Figures 4.15-4.18 illustrate the Laser Raman spectra of AP-SWNT sample treated by different TETA volume and sonication times. The Raman spectra results indicate the AP-SWNT sample after TETA treatment having the same carbon structure. This is insignificant effect of sonication on both the percentage carbon purity and the carbon structure.

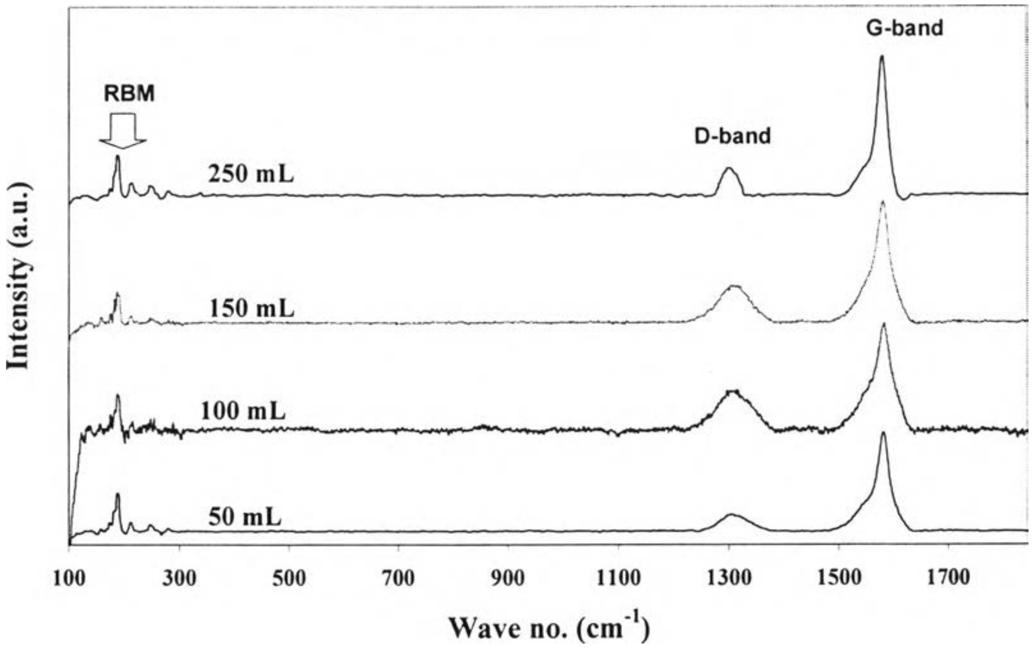

Figure 4.15 Raman spectra of AP-SWNT sample treated with different TETA volumes at 30 minutes of sonication and 70°C.

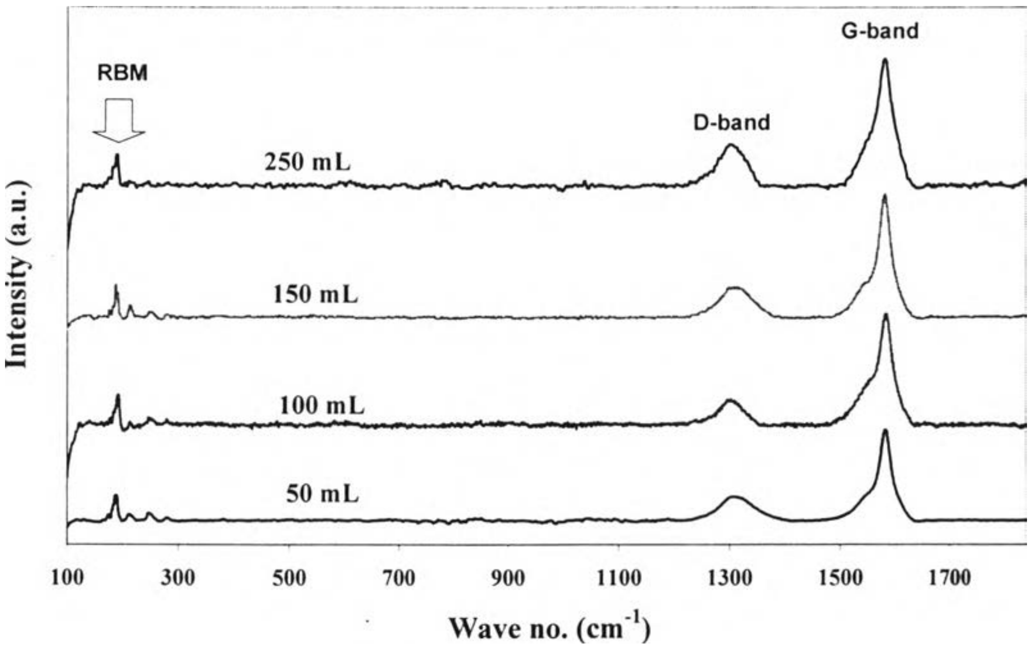

Figure 4.16 Raman spectra of AP-SWNT sample treated with different TETA volumes at 1 hour of sonication and 70°C.

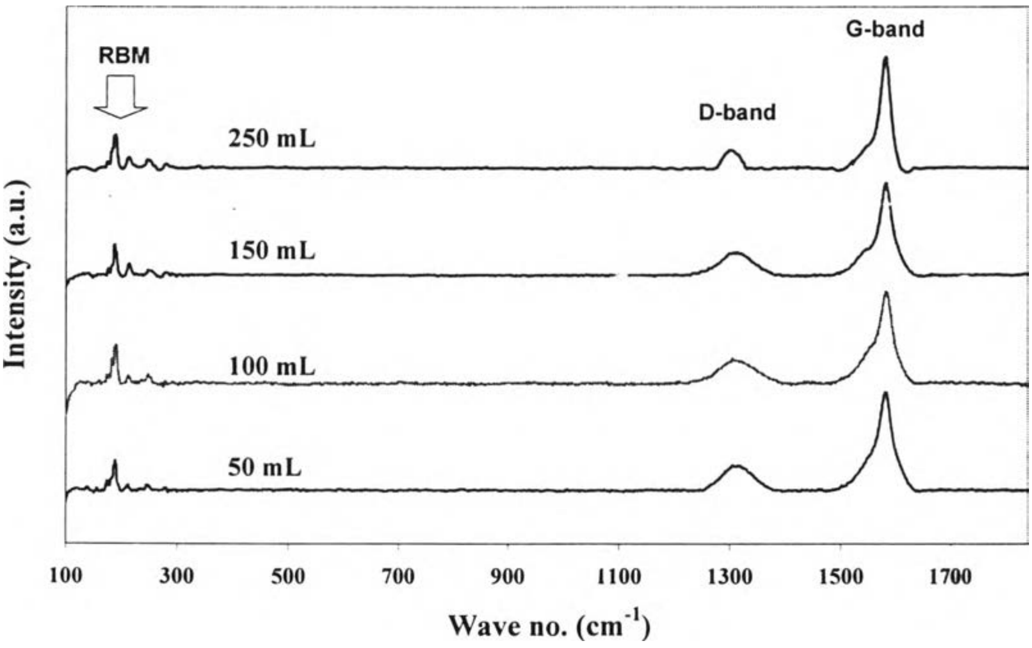

Figure 4.17 Raman spectra of AP-SWNT sample treated with different TETA volumes at 3 hours of sonication and 70°C.

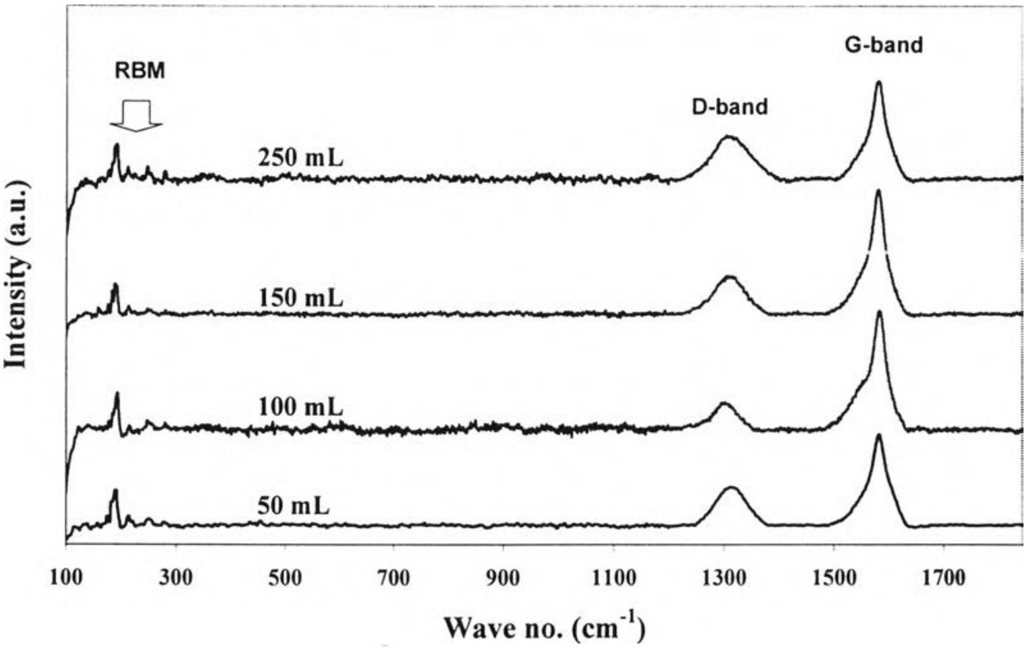

Figure 4.18 Raman spectra of AP-SWNT sample treated with different TETA volumes at 7 hours of sonication and 70°C.

In order to study the effect of sonication time, the experiments were run at different sonication times of 30 minutes, 1 hour, 3 hours and 7 hours in each TETA volume. The Laser Raman spectroscopy and TPO techniques were used to characterize the samples before and after the froth flotation step.

As shown in Figure 4.19, for any given TETA volume used for the silica dissolution step, an increase in sonication time results in increasing carbon purity. This is because sonication can break partially the carbon deposit on the silica surface leading to TETA to contact the silica surface. As a result, the silica dissolution is increased.

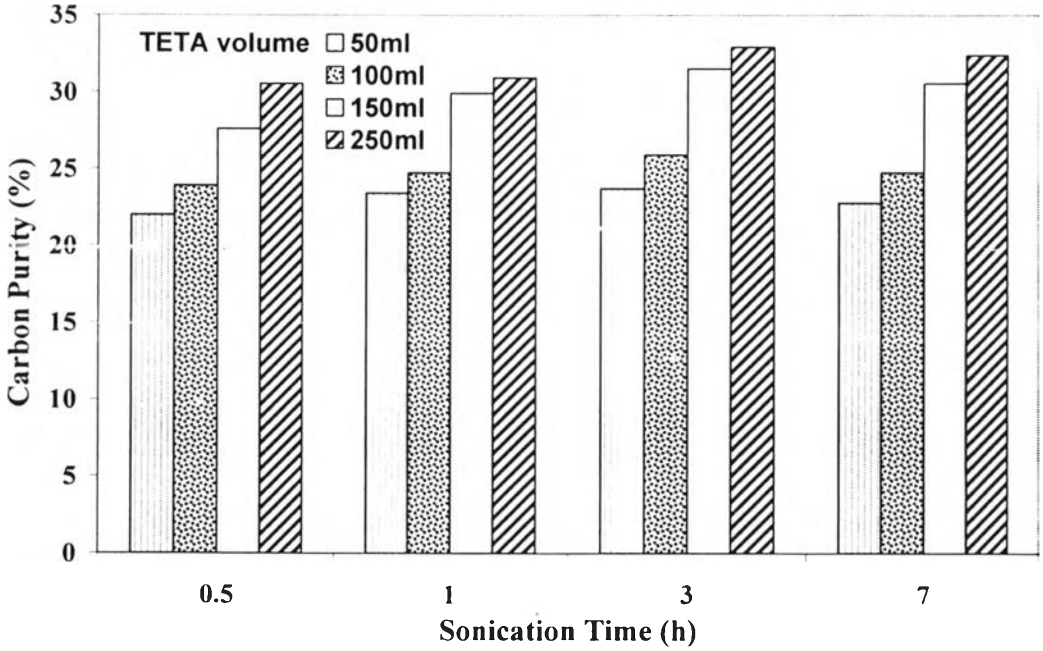

Figure 4.19 Effect of sonication time on the percentage of carbon purity at different TETA volumes and 70°C.

Figures 4.20-4.23 show the Raman spectra of the AP-SWNT samples treated with different TETA volumes and sonication times. All peaks of G-band, D-band and RBM were found in all samples indicated the carbon structure is not destroyed by the TETA treatment.

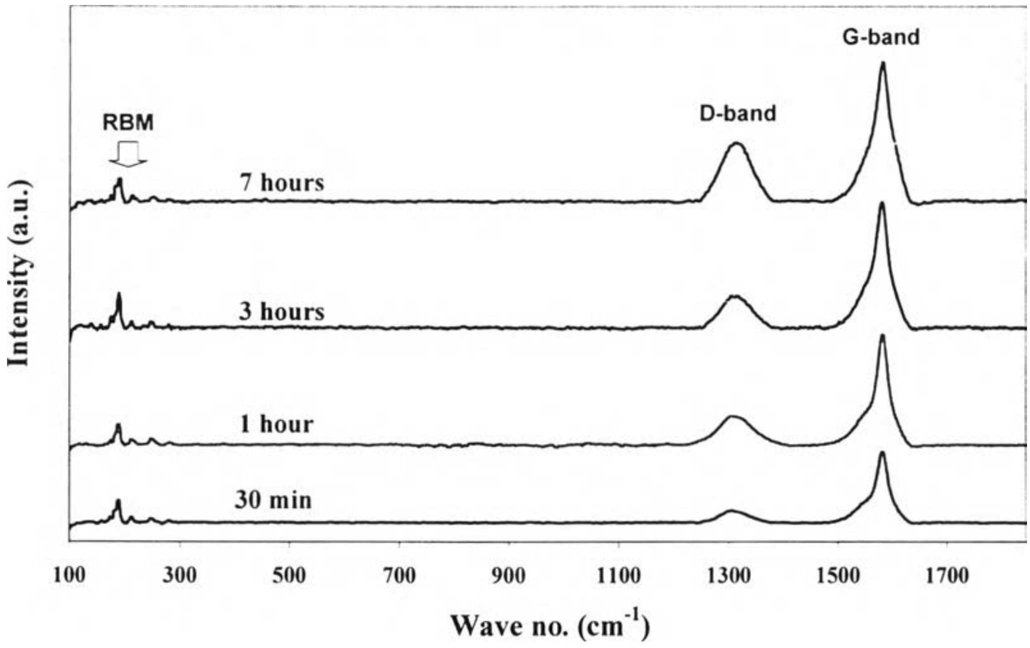

Figure 4.20 Raman spectra of AP-SWNT samples after TETA treatment of 50 mL at different sonication and 70°C.

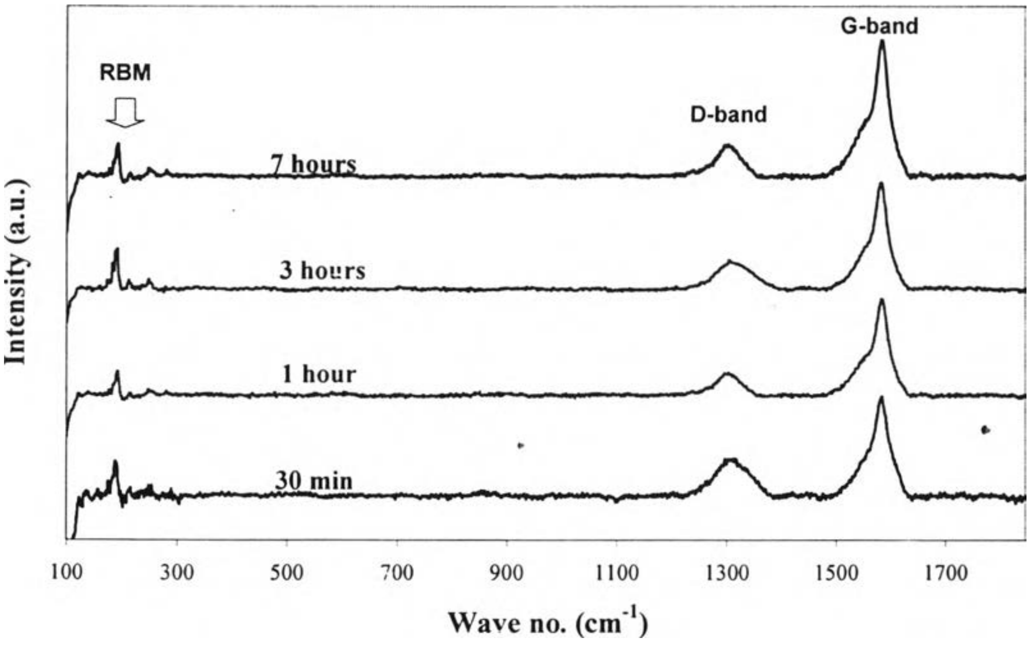

Figure 4.21 Raman spectra of AP-SWNT samples after TETA treatment of 100 mL at different sonication and 70°C.

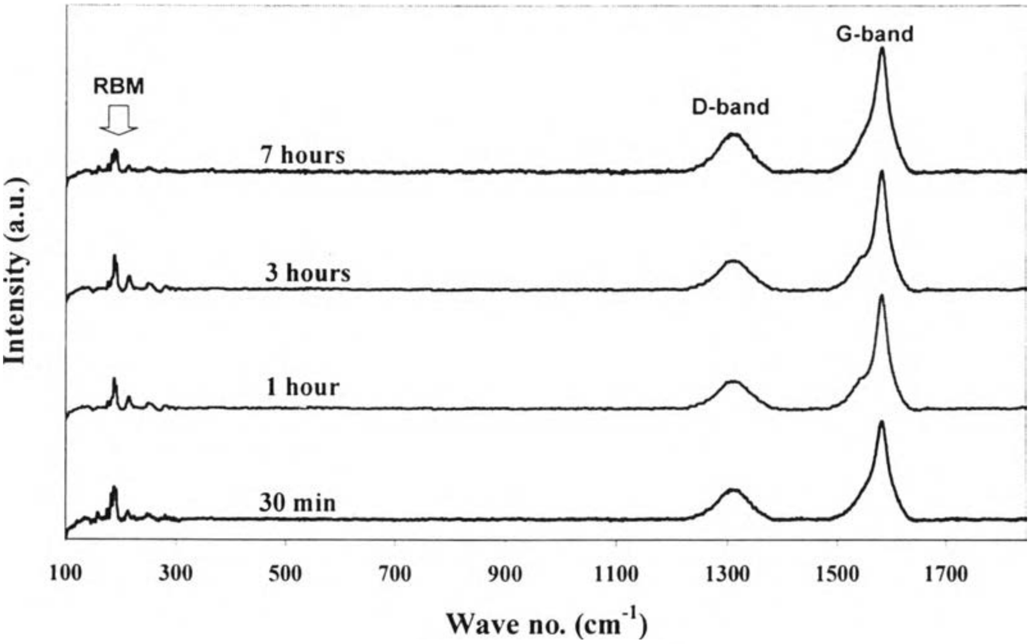

Figure 4.22 Raman spectra of AP-SWNT samples after TETA treatment of 150 mL at different sonication and 70°C.

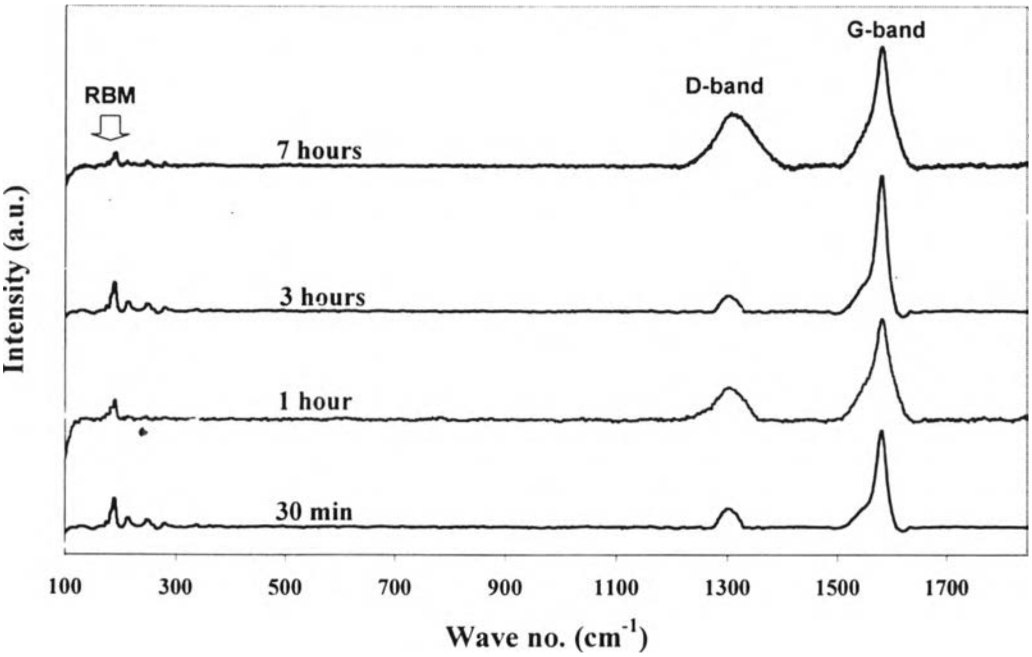

Figure 4.23 Raman spectra of AP-SWNT samples after TETA treatment of 250 mL at different sonication and 70°C.

4.2.1.2 Sonication with NaOH pretreatment

Table 4.7 summarizes the results of NaOH pretreatment at 70°C and 3 h of sonication. As show in Figure 4.24 and Table 4.7, the percentage of carbon purity increases with increasing NaOH concentration and it reaches the maximum value about 56% when the NaOH concentration exceeds 5 M. As compared to the dissolution of pure silica, silica was found to be dissolved completely at 1 M NaOH concentration (see Figure 4.4). The results indicate that some silica particles had complete coverage of the froth of SWNTs.

Table 4.7 Summary of carbon purity of AP-SWNTs after NaOH pretreatment at different NaOH concentrations, 70°C and 3 h of sonication

NaOH concentration	Carbon Purity (%)	RBM
0.5 M	28.19	yes
1.0 M	53.48	yes
3.0 M	55.48	yes
5.0 M	56.03	yes
7.0 M	56.04	yes
10.0 M	56.29	yes

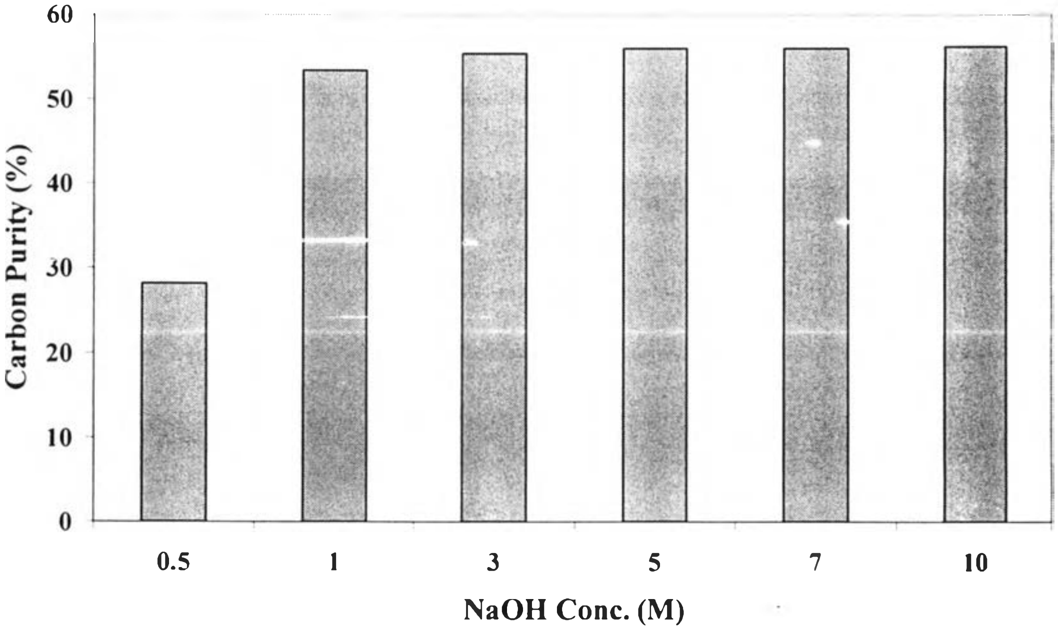

Figure 4.24 Effect of NaOH concentration on the carbon purity of AP-SWNTs after NaOH pretreatment at 70°C and 3 hours of sonication.

Figure 4.25 shows the Laser Raman results of AP-SWNT samples after NaOH pretreatment. The RBM peak corresponding to the appearance of SWNTs was found in all samples. Both D and G bands were also found in all samples. The Raman results suggest that NaOH pretreatment does not alter the carbon structure of the AP-SWNTs.

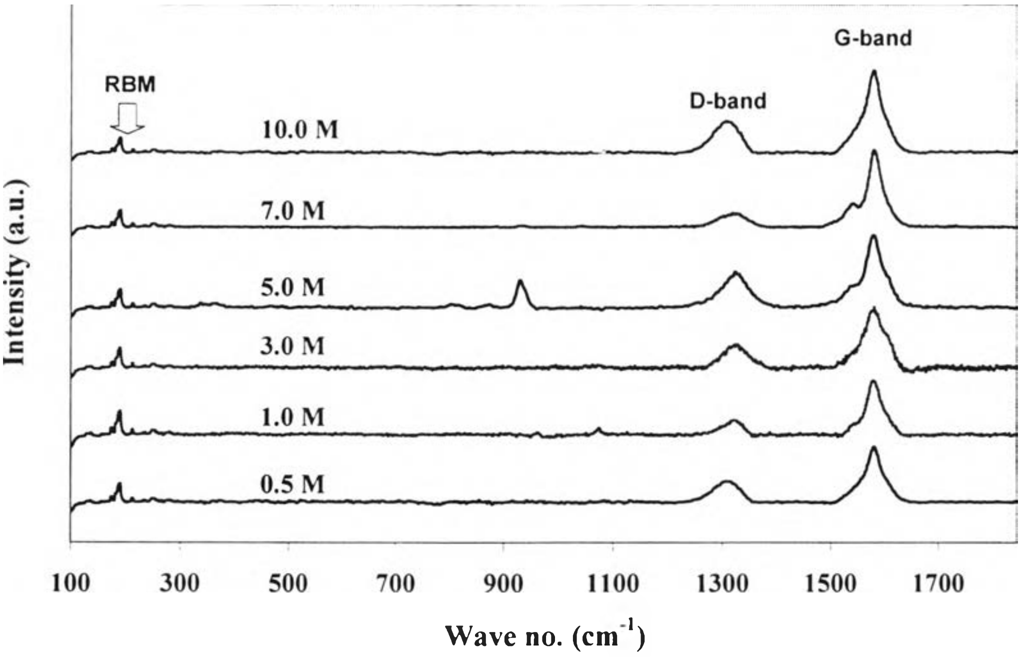

Figure 4.25 Raman spectra of AP-SWNT samples after NaOH pretreatment at different NaOH concentrations, 70°C and 3 hours of sonication.

4.2.2 Froth flotation Results

4.2.2.1 Froth flotation with TETA pretreatment

As described before, AP-SWNT samples were primarily treated by either TETA or NaOH with sonication before running froth flotation. The froth flotation experiments were terminated when no foam came out from the flotation column. Table 4.8 summarizes all of the results of the combined treatment of TETA and froth flotation. The Raman spectra of carbon products after sonication with distilled water for 3 hours and froth flotation, is shown in Figure 4.26. The D band was smaller than the G band indicating that the carbon products contained mostly SWNTs more than amorphous carbons. The result is also confirmed by the TPO profiles as shown in Figure 4.27. The amount of carbon is calculated from the area of the TPO peaks. As shown in Table 4.8, for a case of distilled water, the carbon purity of the carbon products is 10.62% as compared to 4% of the AP-SWNT feed solution. Both TETA treatment and sonication could enhance the process performance of froth flotation in terms of carbon purity of the carbon products obtained from the collapsed froth. More discussion of the results will be given in the next paragraph.

Table 4.8 Summary of purity of AP-SWNTs after TETA treatment with sonication and froth flotation under different operational conditions

Operational Conditions	Carbon Purity (%)	RBM
50mL Di-water, 3 hours	10.62	yes
50mL TETA l, 30 minutes	27.54	yes
50mL TETA, 1 hour	30.07	yes
50mL TETA, 3 hours	39.06	yes
50mL TETA, 7 hours	35.23	yes
100mL TETA, 30 minutes	28.88	yes
100mL TETA, 1 hour	33.39	yes
100mL TETA, 3 hours	40.44	yes
100mL TETA, 7 hours	38.09	yes
150mL TETA, 30 minutes	30.88	yes
150mL TETA, 1 hour	42.60	yes
150mL TETA, 3 hours	51.59	yes
150mL TETA, 7 hours	46.51	yes
250mL TETA, 30 minutes	32.88	yes
250mL TETA, 1 hour	47.60	yes
250mL TETA, 3 hours	55.67	yes
250mL TETA, 7 hours	51.78	yes

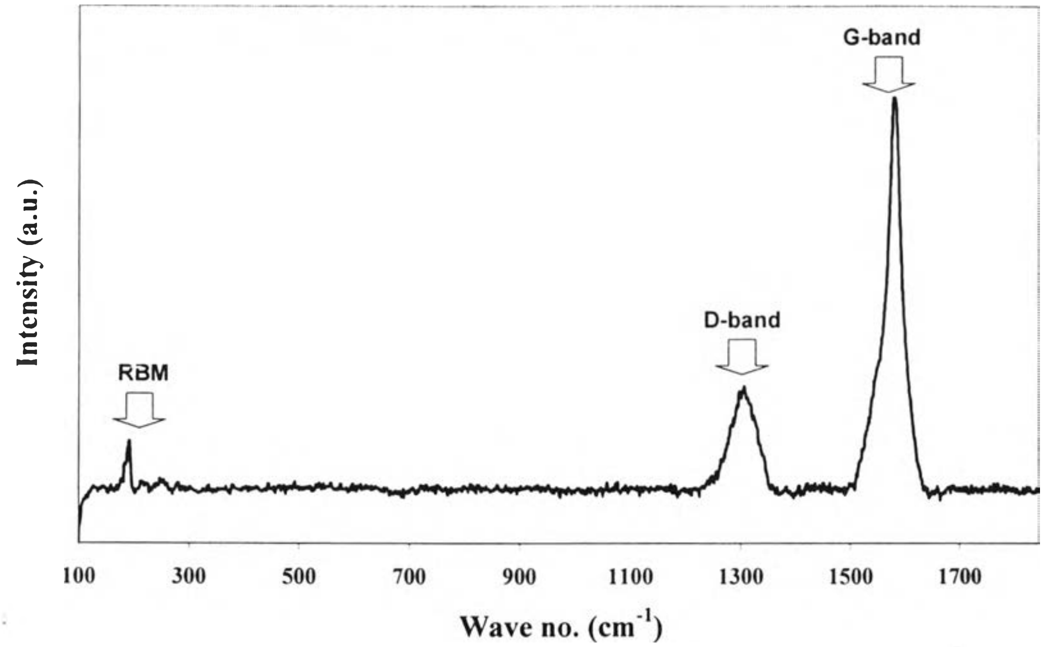

Figure 4.26 Raman spectra of carbon products after sonication with distilled water for 3 hours at 70°C and froth flotation.

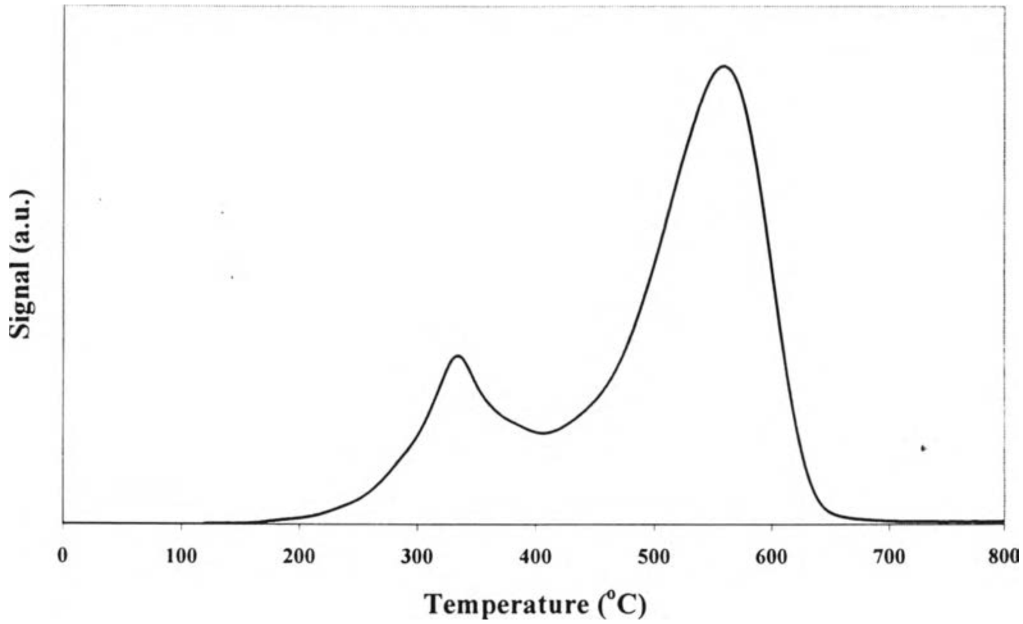

Figure 4.27 TPO profile of carbon products after sonication with distilled water for 3 hours at 70°C and froth flotation.

Figure 4.28 shows the effects of TETA volume and sonication time on the performance of froth flotation in terms of carbon purity. An increase in either TETA volume and sonication time increased the percentage of carbon purity of carbon products obtained from the collapsed froth come out from the flotation column. This is because the more TETA quantity, the more silica is dissolved. Sonication also can break the bond between SWNTs and the silica surface. Consequently, the percentage of carbon purity of the carbon products in the collapsed froth increases. From the results shows in Table 4.8 and Figure 4.28, the effect of TETA on the separation efficiency of froth flotation operation is much higher than that of sonication.

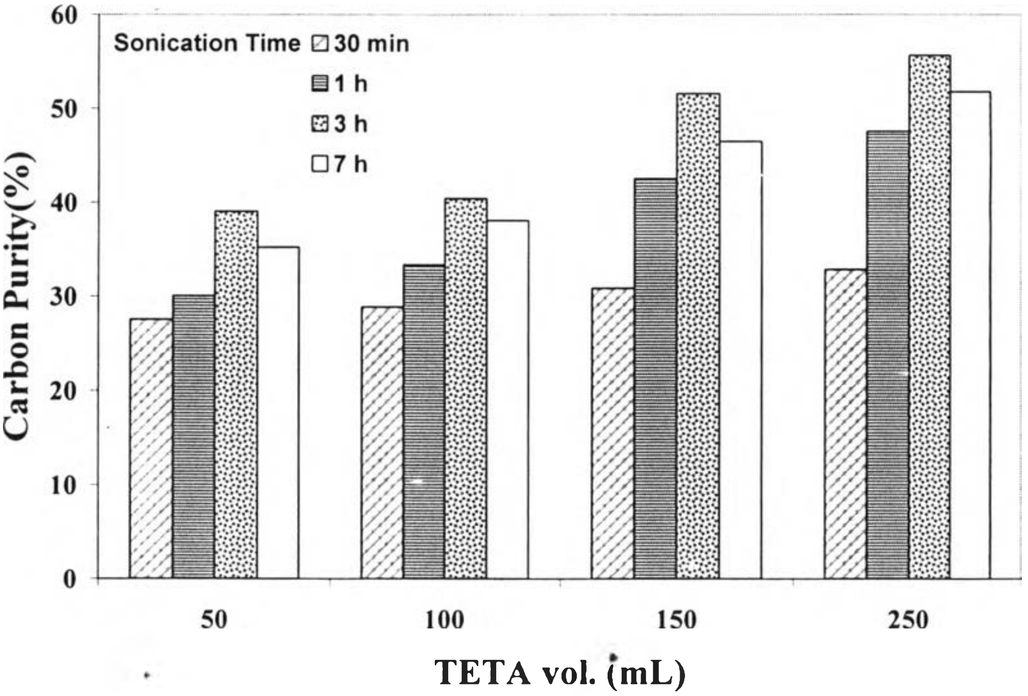

Figure 4.28 Percentage of carbon purity of carbon products obtained from the froth flotation operation with pretreatment of different TETA quantities and different sonication times at 70°C.

Figures 4.29-4.32 illustrate the Laser Raman results of carbon products in the collapsed foams from the froth flotation operation with pretreatment of different TETA volumes and different sonication times at 70°C. The RBM peak corresponding to the appearance of SWNTs was found in all of the carbon product samples. The intensity ratio of D-band to G-band (D/G) provides a relative amount of disordered carbon to graphite-like carbon. From the Laser Raman results, the relative intensity of D-band was still significantly lower than that of G-band indicting that both the pretreatment of TETA with sonication and froth flotation operation do not destroy the structure of SWNTs.

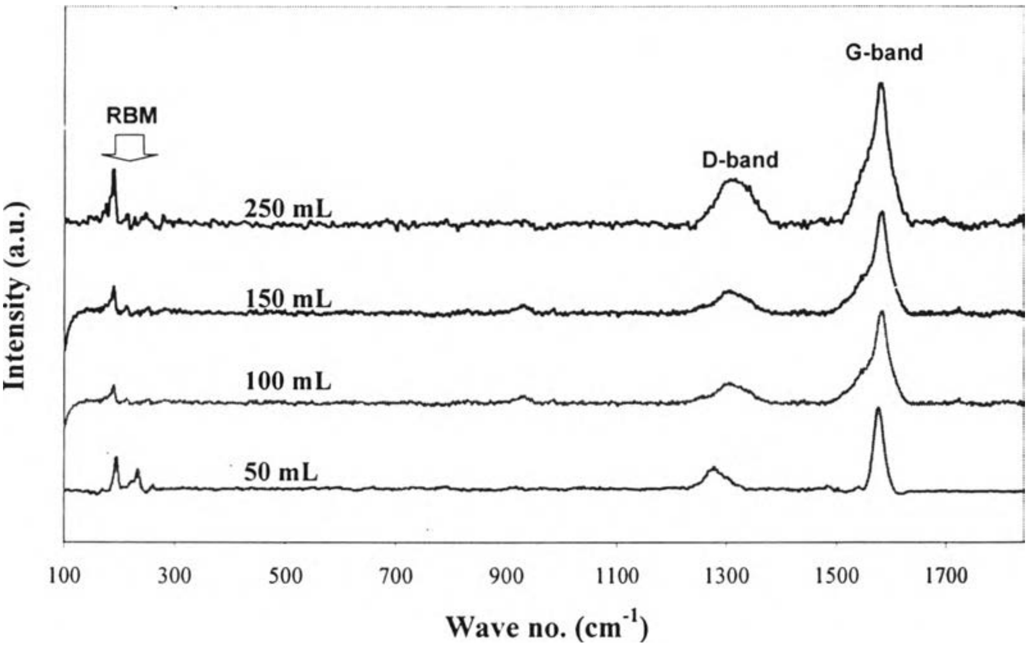

Figure 4.29 Raman spectra of carbon products in the collapsed froth produced from froth flotation operation with pretreatment of various TETA volumes at 70°C and a sonication time of 30 minutes.

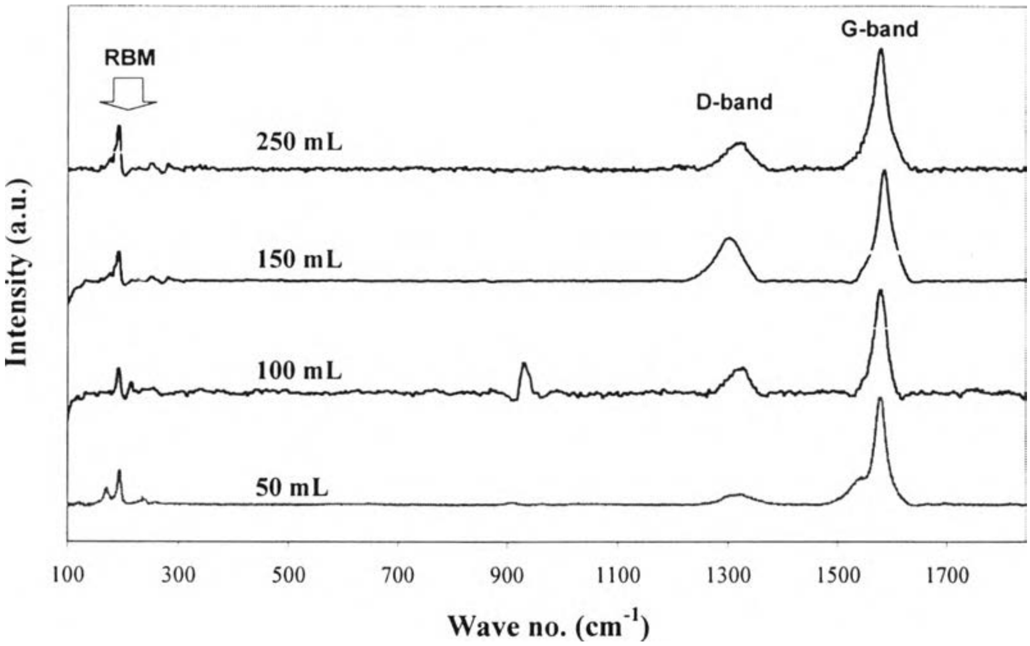

Figure 4.30 Raman spectra of carbon products in the collapsed froth produce from froth flotation operation with pretreatment of various TETA volumes at 70°C and a sonication time of 1 hour.

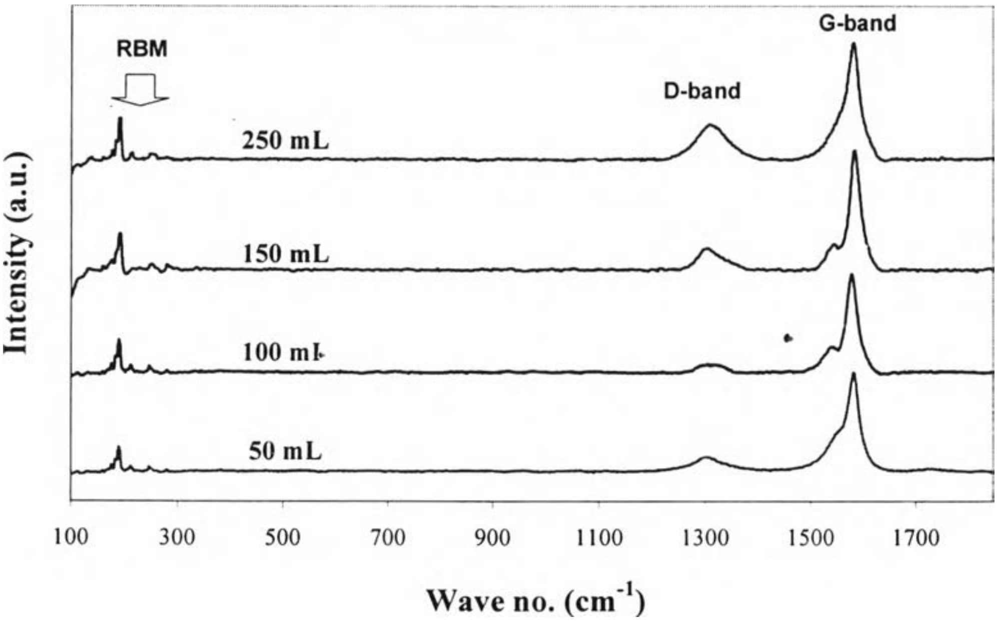

Figure 4.31 Raman spectra of carbon products in the collapsed froth produce from froth flotation operation with pretreatment of various TETA volumes at 70°C and a sonication time of 3 hours.

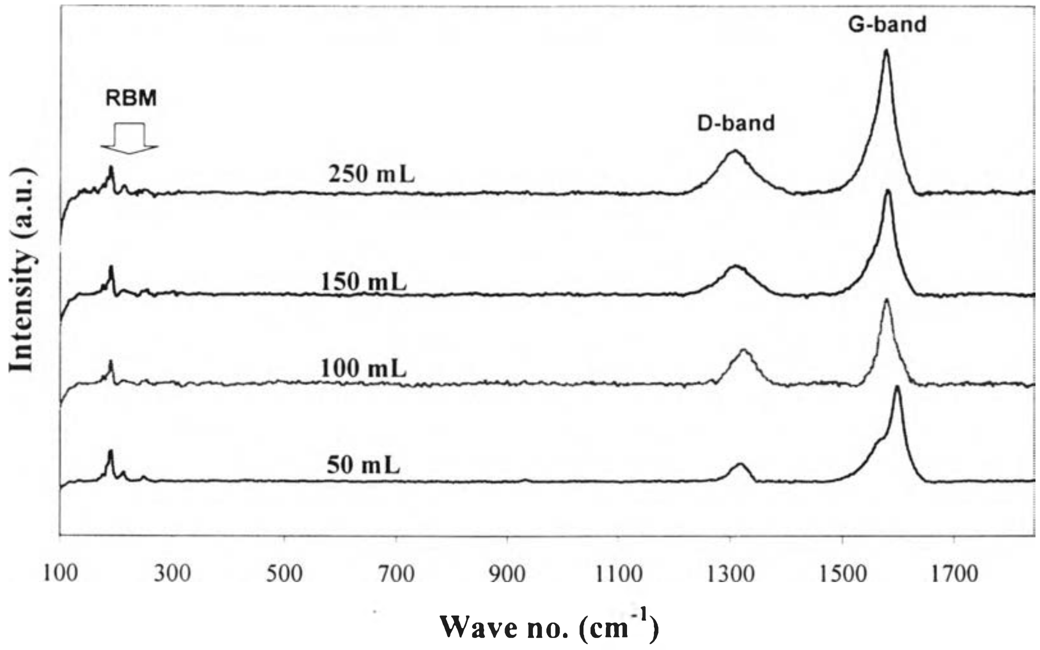

Figure 4.32 Raman spectra of carbon products in the collapsed froth produce from froth flotation operation with pretreatment of various TETA volumes at 70°C and a sonication time of 7 hours.

Figure 4.33 to 4.36 show the effects of TETA volume and sonication time on carbon purity before and after froth flotation step. Interestingly, the more TETA volume and the longer sonication time, the higher carbon purity after the froth flotation step was obtained. The results will be discussed in detail in the next section.

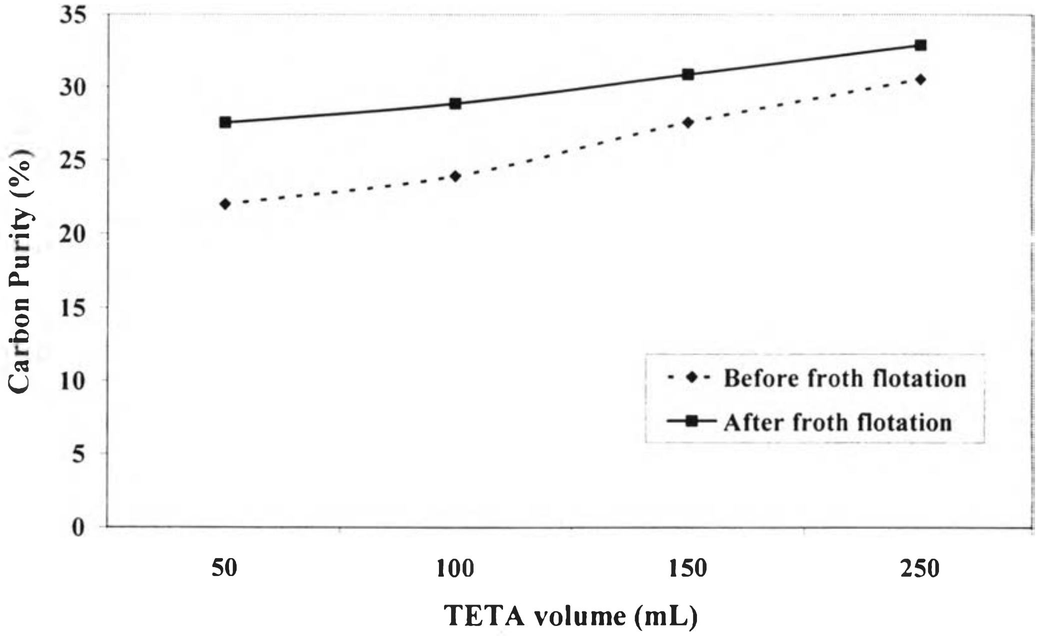

Figure 4.33 Effect of TETA volume on carbon purity before and after froth flotation step at 30 minutes sonication and 70°C.

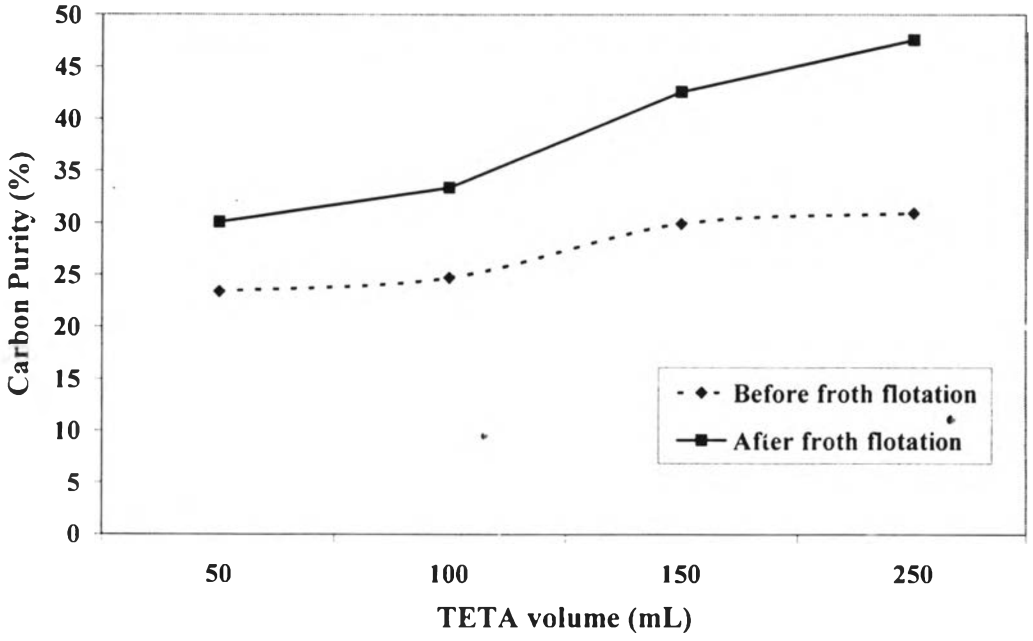

Figure 4.34 Effect of TETA volume on carbon purity before and after froth flotation step at 1 hour sonication and 70°C.

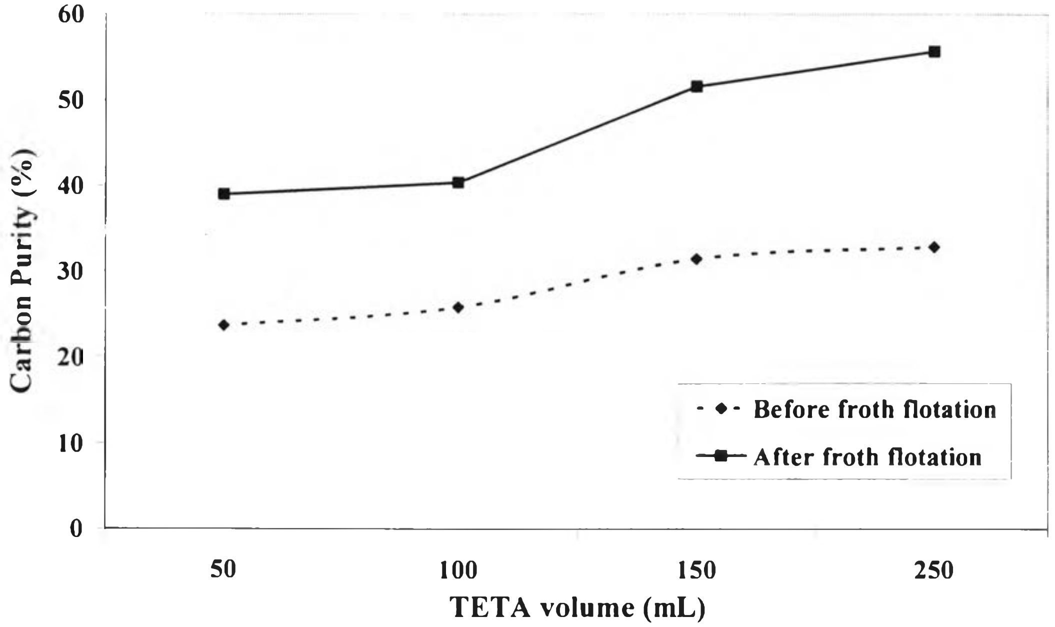

Figure 4.35 Effect of TETA volume on carbon purity before and after froth flotation step at 3 hours sonication and 70°C.

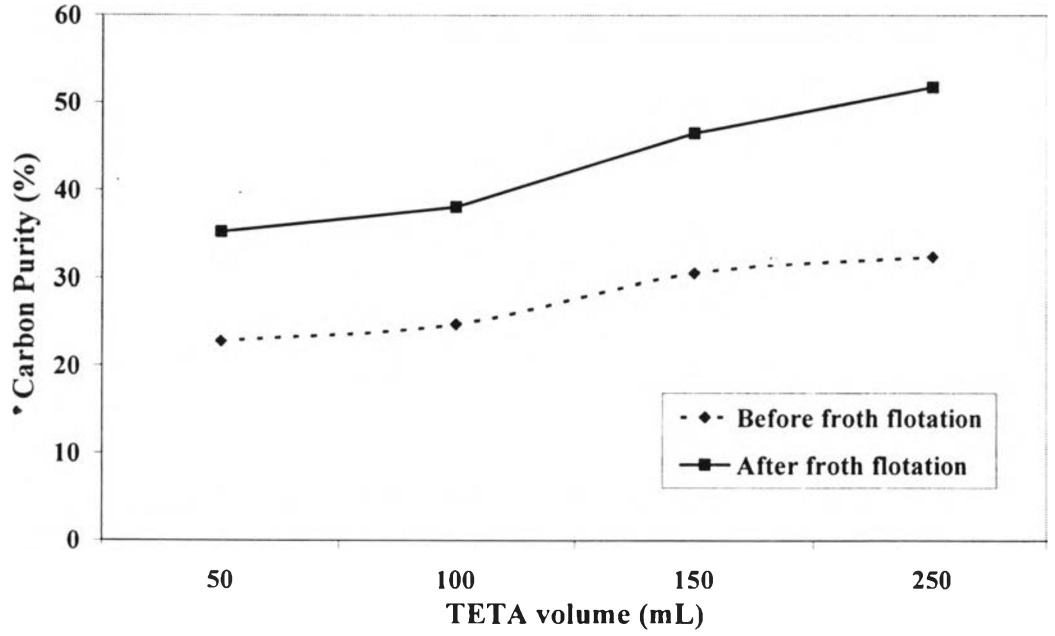

Figure 4.36 Effect of TETA volume on carbon purity before and after froth flotation step at 7 hours sonication and 70°C.

As mentioned before the AP-SWNTs were pretreated with TETA and sonication before running the froth flotation experiments. Figure 4.37 shows the effects of sonication time and TETA volume on the carbon purity of carbon products after the froth flotation step. An increase in either TETA volume or sonication time increased markedly the carbon purity of the carbon products in the collapsed foam from the froth flotation column indicating that sonication can enhance the silica dissolution leading to increasing the flotation efficiency. Interestingly the optimum TETA volume is about 250 mL.

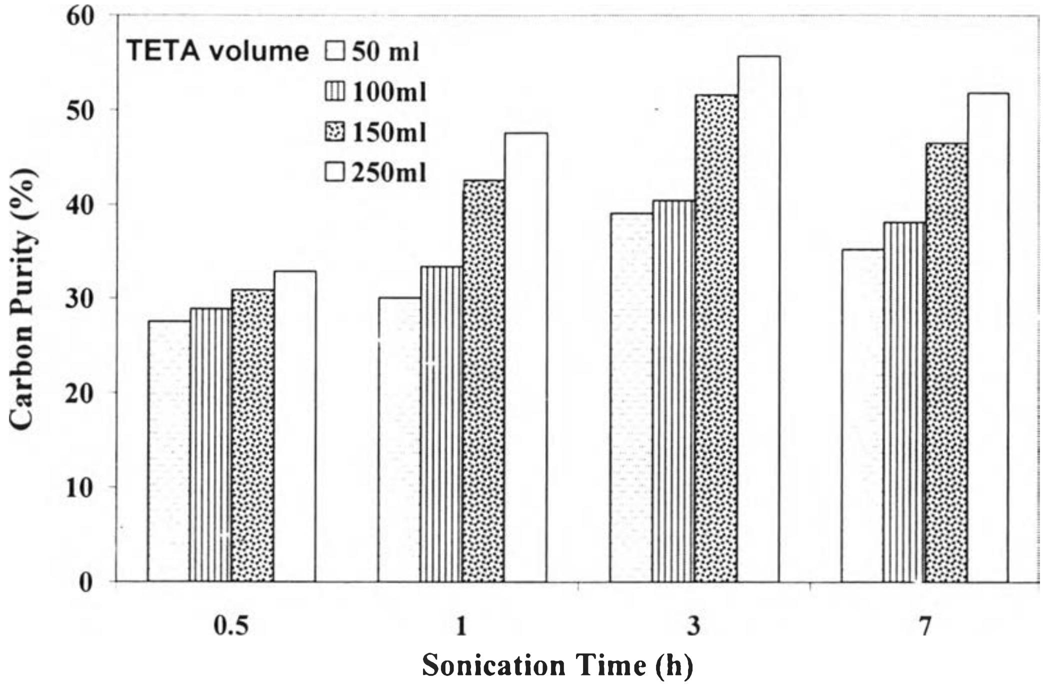

Figure 4.37 Effects of sonication time and TETA volume on the percentage of carbon purity of carbon products after froth flotation step.

Figures 4.38-4.41 show the Raman spectra results of carbon products in the collapsed froth obtained from the froth flotation step having the TETA pretreatment with different TETA volumes and sonication times. From these spectrums, G-band, D-band and the RBM peaks were found in all of samples indicating that the carbon structure is not destroyed by both the TETA treatment and froth flotation. From the Raman results, for any given operational condition the relative intensity of D-band was found to be significantly lower than that of G-band indicating that the formation of amorphous carbon is less than graphite-like carbon.

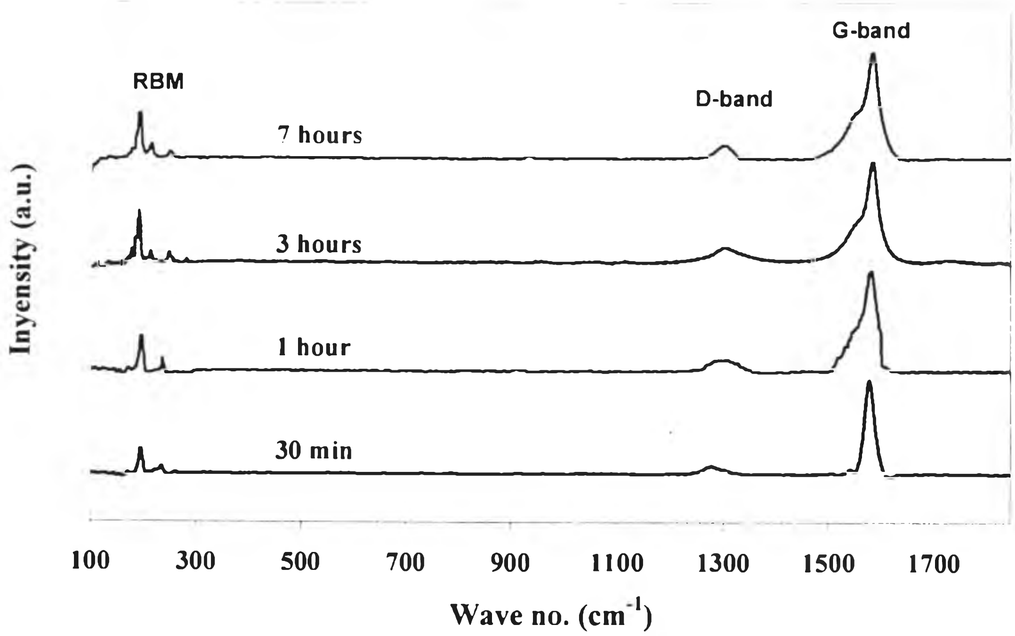

Figure 4.38 Raman spectra of carbon products in the collapsed froth after froth flotation step with TETA pretreatment of 50 mL TETA at different sonication time and 70°C.

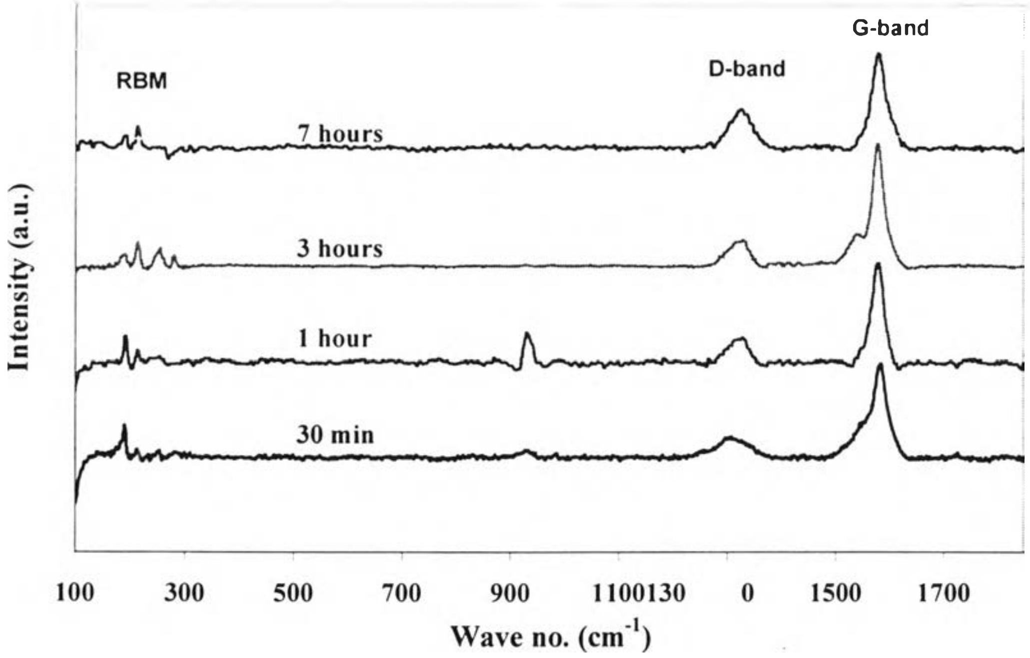

Figure 4.39 Raman spectra of carbon products in the collapsed froth after froth flotation step with TETA pretreatment of 100 mL TETA at different sonication time and 70°C.

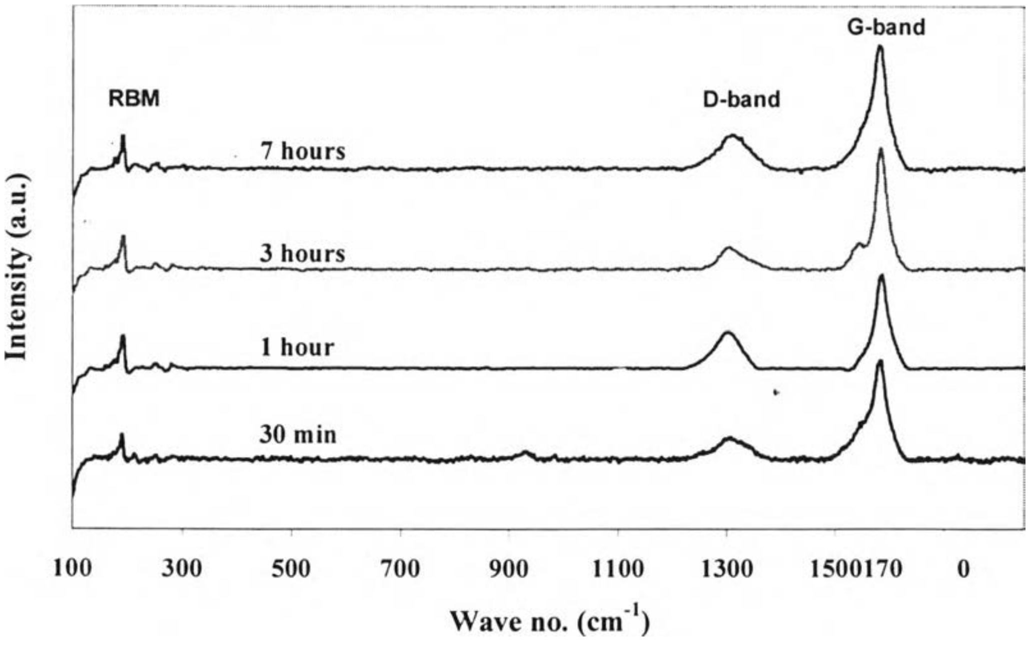

Figure 4.40 Raman spectra of carbon products in the collapsed froth after froth flotation step with TETA pretreatment of 150 mL TETA at different sonication time and 70°C.

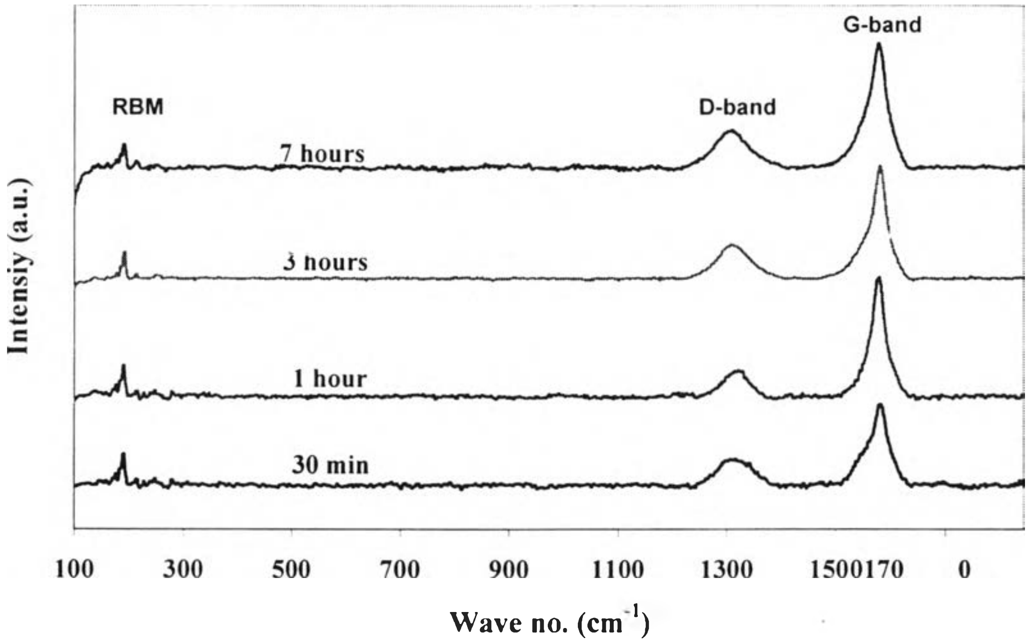

Figure 4.41 Raman spectra of carbon products in the collapsed froth after froth flotation step with TETA pretreatment of 250 mL TETA at different sonication time and 70°C.

Figure 4.42 shows the effects of both TETA volume and sonication time on carbon purity before and after the froth flotation step. For any given conditions of the pretreatment step, the froth flotation could increase approximately two times of the carbon purity. From the results, the maximum carbon purity of 55.67% was achieved corresponding to the optimum TETA volume of 250 mL and the optimum sonication time of 3 hours.

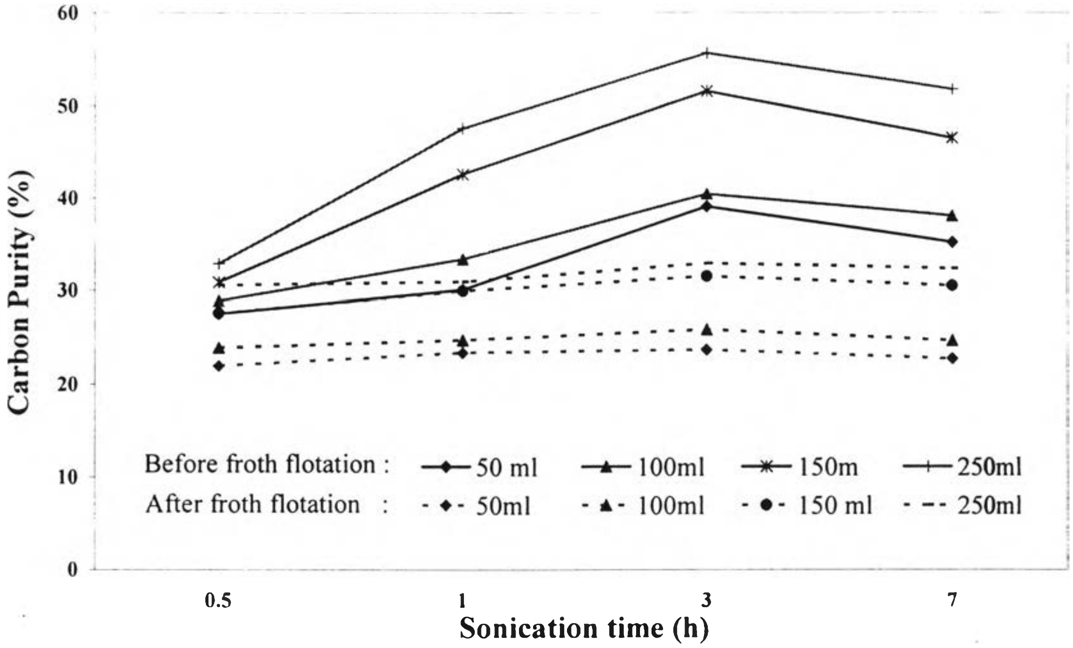

Figure 4.42 Comparison of carbon purity before and after froth flotation at different TETA volumes and sonication times.

4.2.2.2 Froth flotation with NaOH pretreatment

Table 4.9 and Figure 4.43 show the results in terms of carbon purity od SWNTs after froth flotation with NaOH pretreatment at different NaOH concentrations. As can be seen in Figure 4.43, the percentage of carbon purity increases with increasing NaOH concentration when the NaOH concentration is lower than 1 M. Beyond 1.0 M NaOH concentration, the carbon purity decreased slightly with increasing NaOH concentration. The deterioration of carbon purity with increasing NaOH concentration is due to an increase in solution pH. The higher pH, the higher negative charge on the SWNT surface is. Since surfonic L 24-7, nonionic surfactant used in this experiment, a less amount of SWNTs is carried over with froth if a solution pH is increased. Figure 4.44 shows the effect of NaOH concentration used in the pretreatment step on the carbon purity of AP-SWNTs before and after froth flotation. From the figure, the optimum NaOH concentration was found to be 1 M for the maximum carbon purity of 77.74%.

Table 4.9 Carbon purity of AP-SWNT samples after troth flotation with NaOH pretreatment at different NaOH concentrations, 70°C and 3 hours of sonication

NaOH concentration	Carbon Purity (%)	RBM
0.5 M	45.44	yes
1.0 M	72.74	yes
3.0 M	70.53	yes
5.0 M	67.48	yes
7.0 M	65.51	yes
10.0 M	62.74	yes

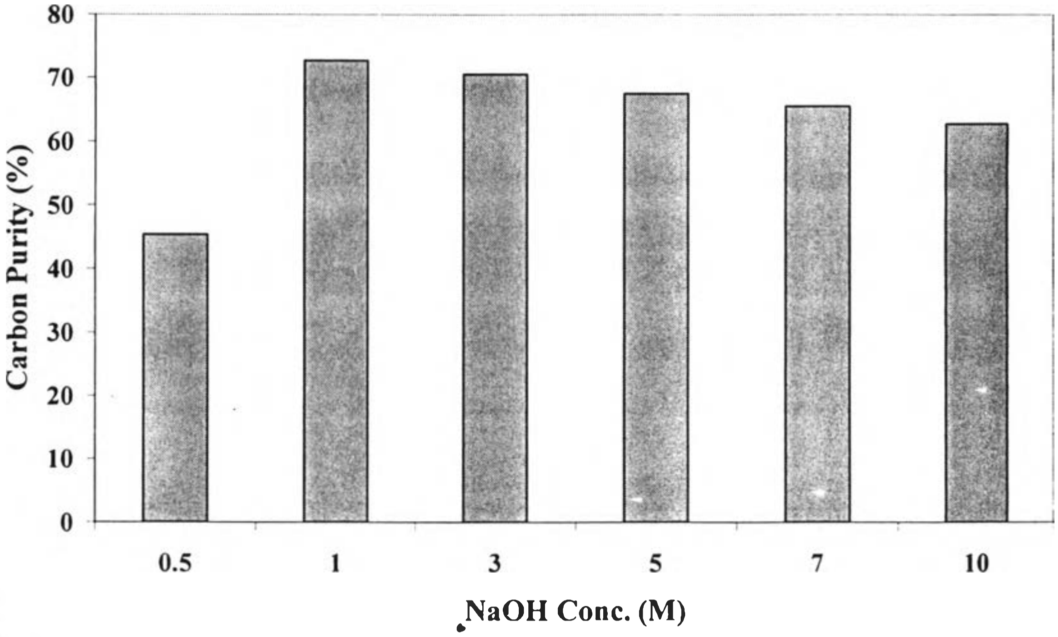

Figure 4.43 Effect of NaOH concentration on the carbon purity of SWNT samples after froth flotation with NaOH pretreatment at 70°C and 3 hours of sonication.

Figure 4.45 shows the Raman results after froth flotation with pretreatment of different NaOH concentration. All RBM peak and D and G bands were found in all of the samples. The relative ration of D-band to G-band was relatively the same for all of the samples. The Raman results indicate that both NaOH pretreatment and froth flotation do not change the structure of AP-SWNTs.

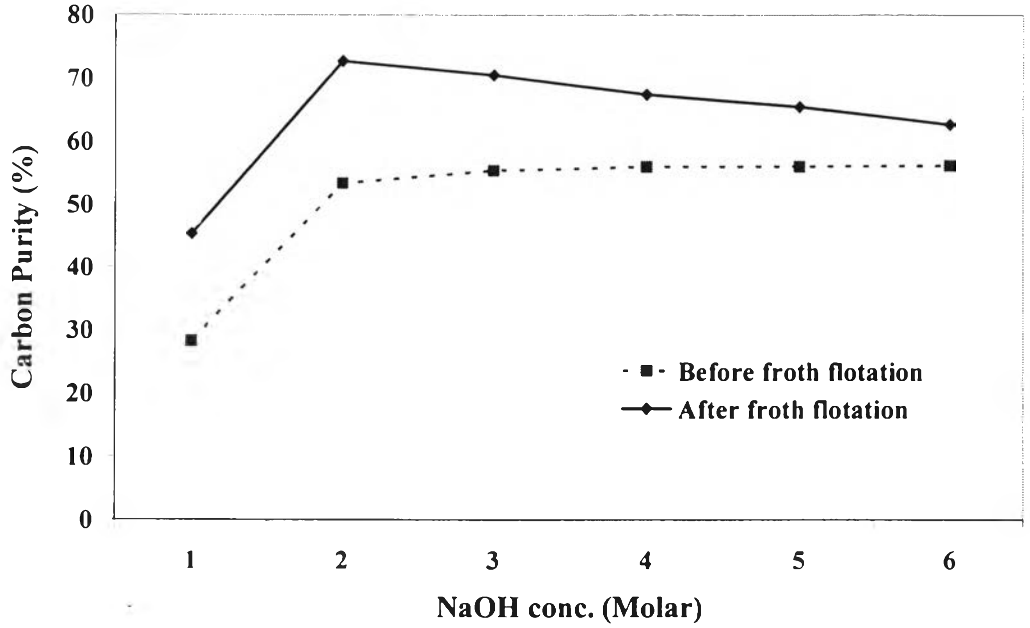

Figure 4.44 Comparison of carbon purity of AP-SWNTs after and before froth flotation with NaOH pretreatment at different NaOH concentrations, 70°C and 3 hours of sonication.

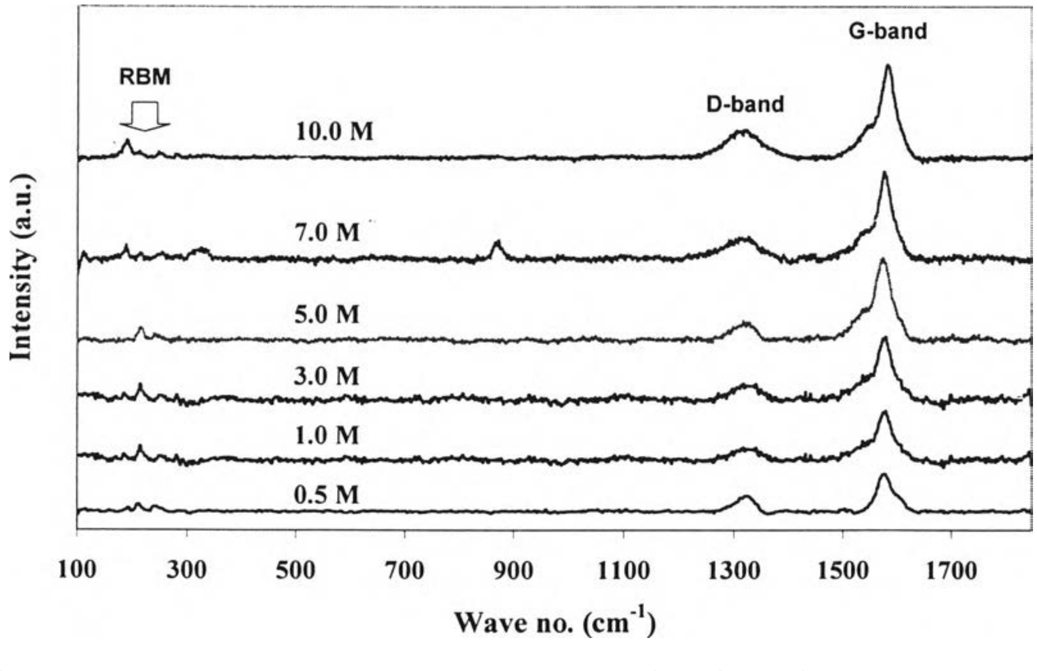

Figure 4.45 Raman spectra of AP-SWNTs after froth flotation with NaOH pretreatment at different NaOH concentrations, 70°C and 3 hours of sonication.