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APPENDICES

Appendix A Centrifugation Techniques Calculation

A.1 Separation of Particles based on Particle Diameter

A particle that is settling under a centrifugal field is governed by two opposing forces which are the centrifugation force and the drag force. Assuming spherical particles, the summation of forces lead to Equation (A.1) (Allen,1981).

$$\sum \text{Force} = (\text{Mass}_p) * (\text{acceleration}) = \text{Centrifugation Force} - \text{Drag Force}$$

$$\frac{\pi}{6}(\rho_p - \rho_f) D^3 \frac{d^2 x}{dt^2} = \frac{\pi}{6}(\rho_p - \rho_f) D^3 \omega^2 x - 3\pi D \mu \frac{dx}{dt} \quad (\text{A.1})$$

Where x = distance from the axis to particle(m)

dx/dt = outward velocity of the particle(m s^{-1})

ρ_p, ρ_f = density of particle and fluid medium, respectively (kg m^{-3})

μ = viscosity of fluid medium ($\text{kg m}^{-1} \text{s}^{-1}$)

D = equivalent spherical diameter of particle (m)

ω = speed of rotation of centrifuge (rad sec^{-1})

At terminal velocity, the summation of these forces equal zero. Thus, Equation (A.1) becomes

$$\frac{dx}{dt} = \frac{1}{18\mu}(\rho_p - \rho_f) D^2 \omega^2 x \quad (\text{A.2})$$

$$\int_S^r \frac{dx}{x} = \int_0^{D_m} \frac{(\rho_p - \rho_f)}{18\mu} D^2 \omega^2 dt$$

$$\ln \frac{r}{S} = \frac{(\rho_p - \rho_f)}{18\mu} D_m^2 \omega^2 t$$

For $r = R$ which is the bottom of the tube, all particles bigger than D_m are centrifuged.

$$D_m = \sqrt{\frac{18\mu \ln(R/S)}{(\rho_p - \rho_f) \omega^2 t}} \quad (\text{A.3})$$

Where S = distance from the axis of rotation to the surface of the fluid (m)

R = distance from the axis of rotation to the bottom of the tube (m)

L = distance from the surface of the fluid to the bottom of the tube (m)

D_m is calculated based on the physical properties of the particle and the fluid, and the applied centrifugation conditions. D_m serves as a guide if the centrifugation conditions used are sufficient to separate the appropriate particles. Figure A.1 shows the configuration of the centrifuges used.

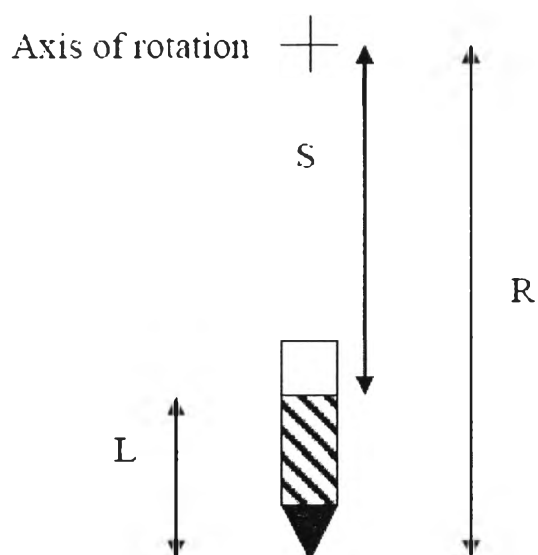


Figure A.1 Configuration of centrifuge used.

Table A.1 Configuration of the centrifuge used

Centrifuge	S(mm)	R(mm)	Centrifuge Tube used
Thermal scientific Sorvall Legend X1R(Fixed Angle Carbon Fiber Rotor (F15-8x50c))	43	104	Fisherbrand centrifuge tube 50 mL
Thermal scientific Sorvall Legend X1R(Round Buckets(TX-400))	68	168	Fisherbrand centrifuge bottle 250 mL
Eppendorf Micro Centrifuge 5415C	39	73	Fisherbrand microcentrifuge 1.5 mL

A.1.1 Crude Oil Preparation

Centrifuge tube (50 mL) is used to prepare crude oil at 14000 x gforce for 3 hours. Therefore, the minimum particle sizes (D_m) that can separate out using this condition.

$$D_m = \sqrt{\frac{18\mu \ln(R/S)}{(\rho_p - \rho_f) \omega^2 t}}$$

$$RCF = gforce = \frac{r\omega^2}{g} \quad \text{and} \quad \omega = \frac{2\pi rpm}{60}$$

$$RCF = 1.12r \left(\frac{rpm}{1000} \right)^2$$

$$14000 = 1.12(104) \left(\frac{rpm}{1000} \right)^2$$

$$rpm = 10963.23 \text{ rpm}$$

$$\text{or } \omega = \sqrt{\frac{RCF \times g}{r}} = \sqrt{\frac{14000 \times 9800}{104}} = 1148.99 \text{ rad/s}$$

$$\therefore D_m = \sqrt{\frac{18\mu \ln(R/S)}{(\rho_p - \rho_f) \omega^2 t}} = \sqrt{\frac{18\mu \ln(104/43)}{(\rho_p - \rho_f) 1148.99^2 (3 \times 60 \times 60)}} = 1.132 \times 10^{-3} \sqrt{\frac{\mu}{(\rho_p - \rho_f)}} \text{ m (A.4)}$$

· If large centrifuge bottle (250 mL) is used to separated clay, sand, and water out from crude oil at 3500 rpm, required time for separating same particle size as centrifuge tube can calculate.

$$\omega = \frac{2\pi rpm}{60} = \frac{2\pi (3500)}{60} = 366.52 \text{ rad/s}$$

From (A.4)

$$\therefore D_m = 1.132 \times 10^{-3} \sqrt{\frac{\mu}{(\rho_p - \rho_f)}} = \sqrt{\frac{18\mu \ln(168/68)}{(\rho_p - \rho_f) 366.52^2 t}}$$

$$t = 34663 \text{ seconds} = 9.63 \text{ hours}$$

Thus, we use the speed at 3500 rpm for 10 hours to pretreat crude oil in this study.

A.1.2 Characterization and Fractionation

Microcentrifuge tube (1.5 mL) is used to centrifuge the precipitated asphaltenes from solution at 14000 rpm for 10 minutes. The minimum diameter of particle can be calculated.

$$\omega = \frac{2\pi \text{ rpm}}{60} = \frac{2\pi (14000)}{60} = 1466.08 \text{ rad/s}$$

$$\therefore D_m = \sqrt{\frac{18\mu \ln(R/S)}{(\rho_p - \rho_f)\omega^2 t}} = \sqrt{\frac{18\mu \ln(73/39)}{(\rho_p - \rho_f)1466.08^2 (10 \times 60)}} = 9.354 \times 10^{-5} \sqrt{\frac{\mu}{(\rho_p - \rho_f)}} \text{ m} \quad (\text{A.5})$$

If large centrifuge bottle (250 mL) is used to centrifuge the precipitated asphaltenes out at 3500 rpm, required time can be calculated.

From (A.5)

$$\therefore D_m = 9.354 \times 10^{-5} \sqrt{\frac{\mu}{(\rho_p - \rho_f)}} \text{ m} = \sqrt{\frac{18\mu \ln(168/68)}{(\rho_p - \rho_f)366.52^2 t}}$$

$$t = 13850 \text{ seconds} = 3 \text{ hours } 50 \text{ minutes}$$

The precipitated asphaltenes is centrifuged at 3500 rpm for 4 hours.

A.2 Concentration of Crude Oil and Amount of Asphaltenes Precipitated

Concentration of heptane in solution can calculate as shown in equation A.6

$$\% \text{ Heptane concentration} = \frac{\frac{m_{\text{heptane}}}{\rho_{\text{heptane}}}}{\frac{m_{\text{heptane}}}{\rho_{\text{heptane}}} + \frac{m_{\text{oil}}}{\rho_{\text{oil}}}} \times 100\% \quad (\text{A.6})$$

$$\text{Mass fraction of crude oil} = m_{\text{oil}} / (m_{\text{oil}} + m_{\text{heptane}}) = x$$

$$\text{Given mass of solution} = m_{\text{sol}}$$

$$\text{mass of asphaltene (after wash and dry in oven)} = m_{\text{asph}}$$

Amount of asphaltene precipitated can calculate as equation A.7

$$\% \text{ g of asphaltene precipitated per g crude oil} = \frac{m_{\text{asph}}}{m_{\text{sol}} \cdot x} \times 100\% \quad (\text{A.7})$$

Appendix B Properties of Solution

B.1 Solubility Parameter of Solution

Solubility parameter of solution can calculate from Eq (B.1) (Hansen, 2000);

$$\delta_{solution} = \sum \psi_i \delta_i \quad (B.1)$$

Where Ψ_i is the volume fraction of each component and δ_i is solubility parameter of each component.

B.2 Viscosity of Solution

The viscosity of solution can calculated from Eq B.2 (Novak, 2003).

$$\ln \mu_{solution} = \sum \psi_i \ln \mu_i - \frac{\Delta G^E}{RT} \quad (B.2)$$

Term $\frac{\Delta G^E}{RT}$ is for non ideal part. If the ideal solution, the Eq B.2 can be written as Eq B.3.

$$\ln \mu_{solution} = \sum \psi_i \ln \mu_i \quad (B.3)$$

Appendix C The Fraction of Soluble and Insoluble Particles in *Cut 1*

The 2 wt% of *cut 1* asphaltenes in toluene was prepared and centrifuged to separate insoluble particle out at 14000 rpm for 10 minutes using Eppendorf Micro Centrifuge 5415C. Total asphaltenes for *cut 1* in toluene is estimated by evaporate toluene from solution. The particles in toluene can be calculated using Eq C.1.

$$\text{Wt \% particle in toluene} = \frac{x}{A - x} \times 100 \% \quad (\text{C.1})$$

Where x is weight of particle cake (g) and A is weight of toluene (g).

The information of total and insoluble particles in *cut 1* asphaltenes are shown in Table C.1.

Table C.1 Total asphaltenes, soluble and insoluble particle for *cut 1*

	Total		Insoluble	
	#1	#2	#1	#2
Solution (g)	0.4544	0.4688	1.2385	1.2628
Cakes (g)	0.0092	0.0097	0.0122	0.0128
Wt % in toluene	2.07	2.11	1.01	1.03
Wt % in toluene (avg)	2.09		1.02	

Therefore, the insoluble and soluble fraction can be calculate;

$$\text{Insoluble fraction} = \frac{\text{insoluble}}{\text{total}} = \frac{1.02}{2.09} = 0.4859$$

$$\text{Soluble fraction} = 1 - 0.4859 = 0.5141$$

Cut 1 consists of 51 % of insoluble particles and 49 % of soluble particles.

The 1.99 wt% of *cut 1* mixed with 2.01 wt% of 75 vol% heptane soluble asphaltenes in toluene. The total concentration is 3.99 wt%. The concentration of asphaltene in toluene is calculated using Eq C.1.

$$\text{Wt \% of total asphaltenes in toluene} = \frac{3.99}{100 - 3.99} \times 100 \% = 4.16 \text{ wt \%}$$

Amount of insoluble particles in solution is shown in Table C.2.

Table C.2 Amount of insoluble particles in 4 wt% solution of *cut 1* and 75 vol% heptane soluble.

	Insoluble	
	#1	#2
Solution (g)	1.2340	1.2441
Cakes (g)	0.0062	0.0063
Wt % in toluene	0.52	0.53
Wt % in toluene (avg)	0.53	

Therefore, the insoluble and soluble fraction can be calculate;

$$\text{Insoluble fraction} = \frac{\text{insoluble}}{\text{total}} = \frac{0.53}{4.16} = 0.1263$$

$$\text{Soluble fraction} = 1 - 0.1263 = 0.8737$$

The solution contains 13 % of insoluble and 87 % of soluble particles.

Appendix D Asphaltene Stabilization

The amount of precipitated asphaltenes for each cut is shown in Table D.1.

Table D.1 Amount of precipitated asphaltenes for 50 vol% heptane in crude oil

Type of asphaltene	%g asph/ g crude oil	Weight fraction(g asph / g total asph)
50 vol% heptane		
- <i>Cut 1</i>	0.2595	0.0798
- <i>Cut 2</i>	0.3236	0.0995
- <i>Cut 3</i>	0.3164	0.0973
- <i>Soluble</i>	2.3537	0.7235
Total	3.2532	1.0001

Given : Total volume = 4 mL

Assume g crude oil = g toluene

Density of toluene = 0.8668 g/mL

∴ total solvent (Toluene) = $m_{\text{total}} = 4 \text{ mL} \times 0.8668 \text{ g/mL} = 3.4672 \text{ g}$

Total asphaltene = $\frac{3.2532 \text{ g asphaltenes}}{100 \text{ g crudeoil}} \times \frac{1 \text{ g crudeoil}}{1 \text{ g toluene}} \times 3.4672 \text{ g toluene} = 0.1128\text{g}$

The amount of asphaltenes that have to prepare is calculated in Table D.2.

Table D.2 Amount of asphaltenes for 50 vol% heptane in toluene

Type of asphaltene	Calculation	g asphaltenes
50 vol% heptane (<i>cut 1</i>)	0.0798 x 0.1128	0.009
50 vol% heptane (<i>cut 2</i>)	0.0995 x 0.1128	0.0112
50 vol% heptane (<i>cut 3</i>)	0.0973 x 0.1128	0.011
50 vol% heptane (<i>soluble</i>)	0.7235 x 0.1128	0.0816

Appendix E Guinier Approximation

The Guinier approximation can be used to obtain a shape independent estimate about radius of gyration of asphaltene particles. The scattering results are used to estimate the size of nanoaggregates by applying Guinier approximation at low q :

$$\ln(I) = \ln(I_0) - \left(\frac{R_g^2 q^2}{3} \right) \quad (\text{E.1})$$

Where I is scattering intensity, I_0 is scattering intensity at $q = 0$, R_g is gyration radius (\AA) and q is scattering vector (\AA^{-1}).

The slope and intercept of data are then used to calculate $-\frac{R_g^2}{3}$ and $\ln(I_0)$, respectively as shown in Figure D.1. The gyration radius is fitted using Guinier approximation when $R_g q$ is less than 1.3. If the scattering results between $\log I$ and $\log q$ do not reach plateau like *cut 1* uncentrifuged as show in Figure 4.16, slope of Guinier approximation curves is too steep (Figure E.1). The steep line of Guinier approximation is the large gyration radius that make $R_g q$ larger than 1.3.

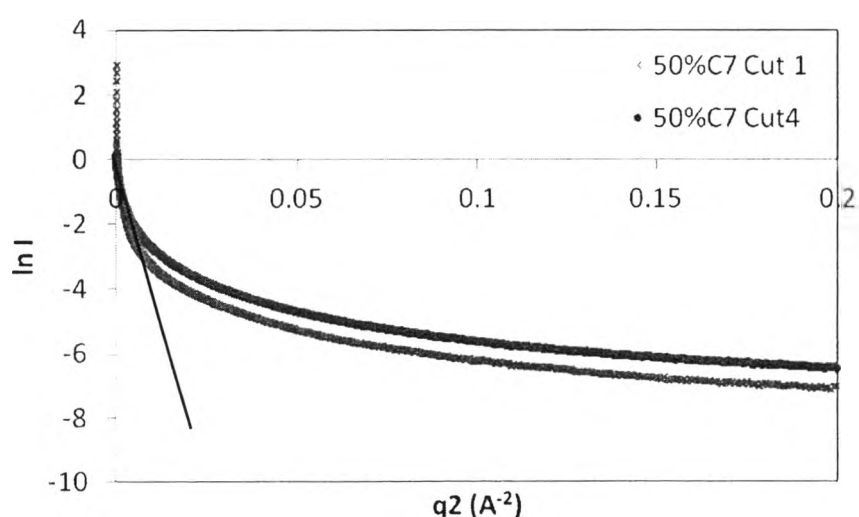
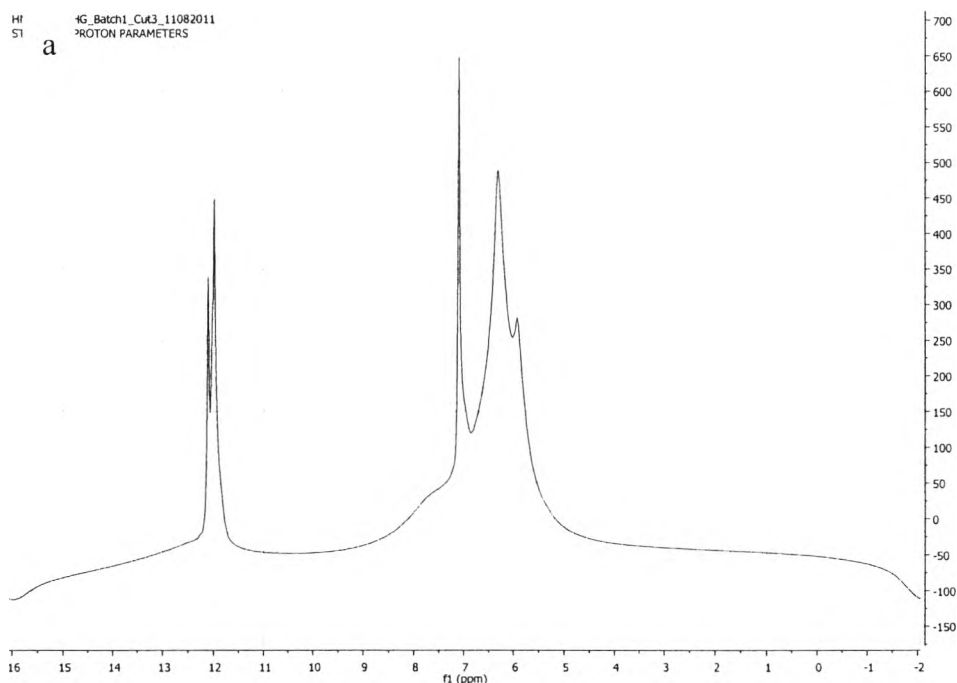


Figure E.1 The Guinier approximation curves.

Appendix F Problem from Nuclear Magnetic Resonance (NMR)

NMR was used to get information about chemical structure of asphaltenes such as their aromaticity and number of carbon per alkyl side chain. First, the asphaltenes were dissolved in deuterated toluene. Figure F.1 shows ^1H NMR and ^{13}C NMR spectrum for *cut 3* asphaltenes in toluene (2 wt%). There are only deuterated toluene peaks for ^1H NMR (2.09, 6.98, 7.00 and 7.09 ppm). Several peaks of deuterated toluene lead to overlap peak with asphaltenes. Therefore, ^1H NMR peaks are not clear and shifted to right axis. It also shows only deuterated toluene peaks for ^{13}C NMR (20.4, 125.49, 128.33, 129.24 and 137.86 ppm). There is no asphaltene peak appear in ^{13}C NMR. There is the possibility that aromatics in asphaltene structures are not soluble in toluene.



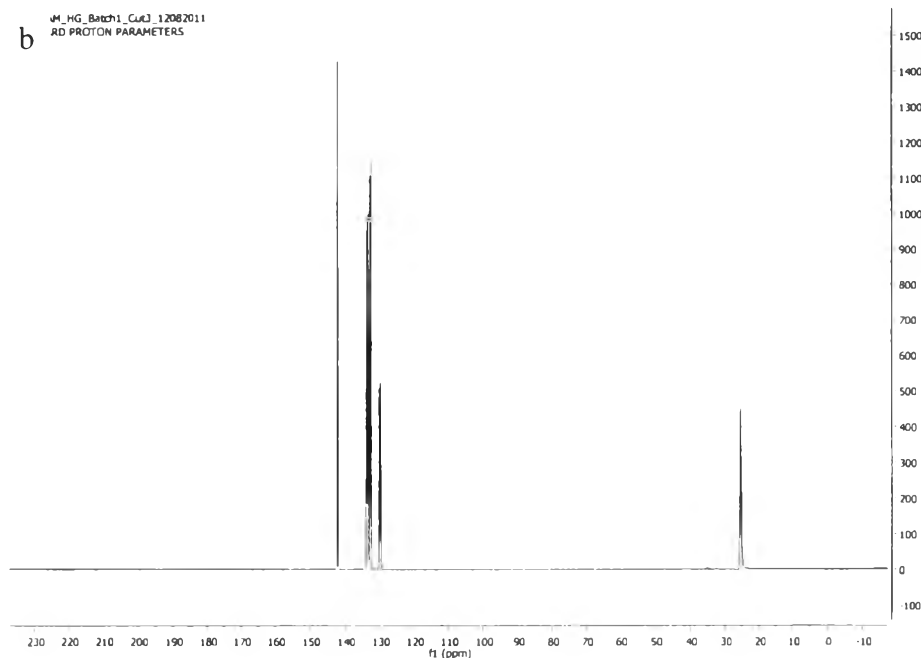


Figure F.1 NMR spectrum of *cut 3* asphaltenes in deuterated toluene (a) ^1H NMR (b) ^{13}C NMR.

The deuterated chloroform was selected as solvent because it is a better solvent for asphaltenes compare to toluene. Moreover, there is only 1 peak for ^1H and ^{13}C NMR at 7.24 and 77.23 ppm, respectively. *Cut 8* is the most stable asphaltenes and have highest solubility due to the lowest heteroatom, metal and aromatic contents. The 75 vol% heptanes soluble asphaltenes (*Cut 8*) was dissolved in deuterated chloroform (1 wt%) to ensure that all of asphaltenes are soluble in solvent. Figure F.2 shows the spectrum of ^1H and ^{13}C NMR for 75 vol% heptane soluble asphaltenes (*Cut 8*) in deuterated chloroform. The ^1H NMR shows only aliphatic peaks of asphaltenes (0-4 ppm). ^{13}C NMR shows only chloroform peak at 77.23 ppm.

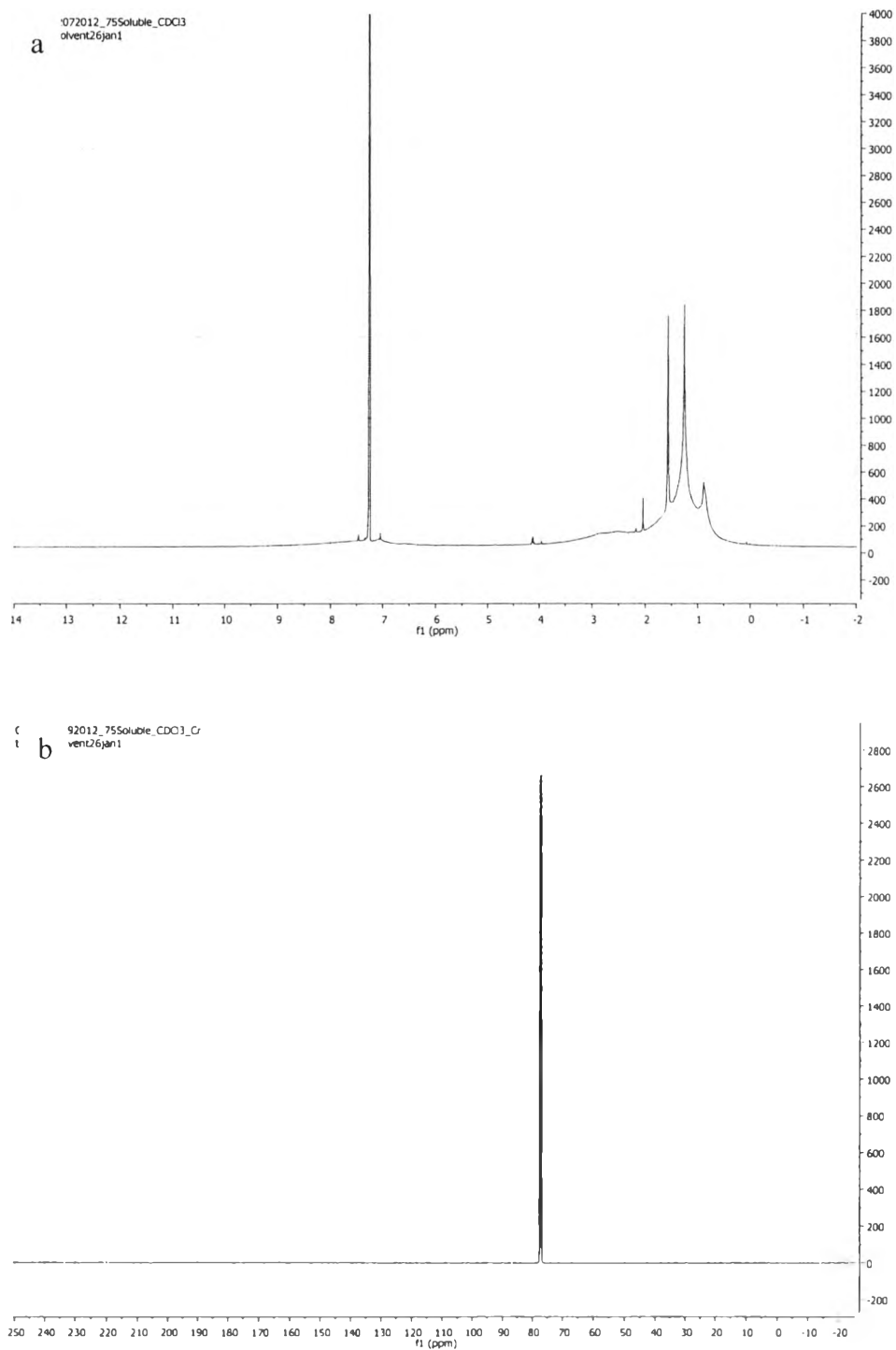
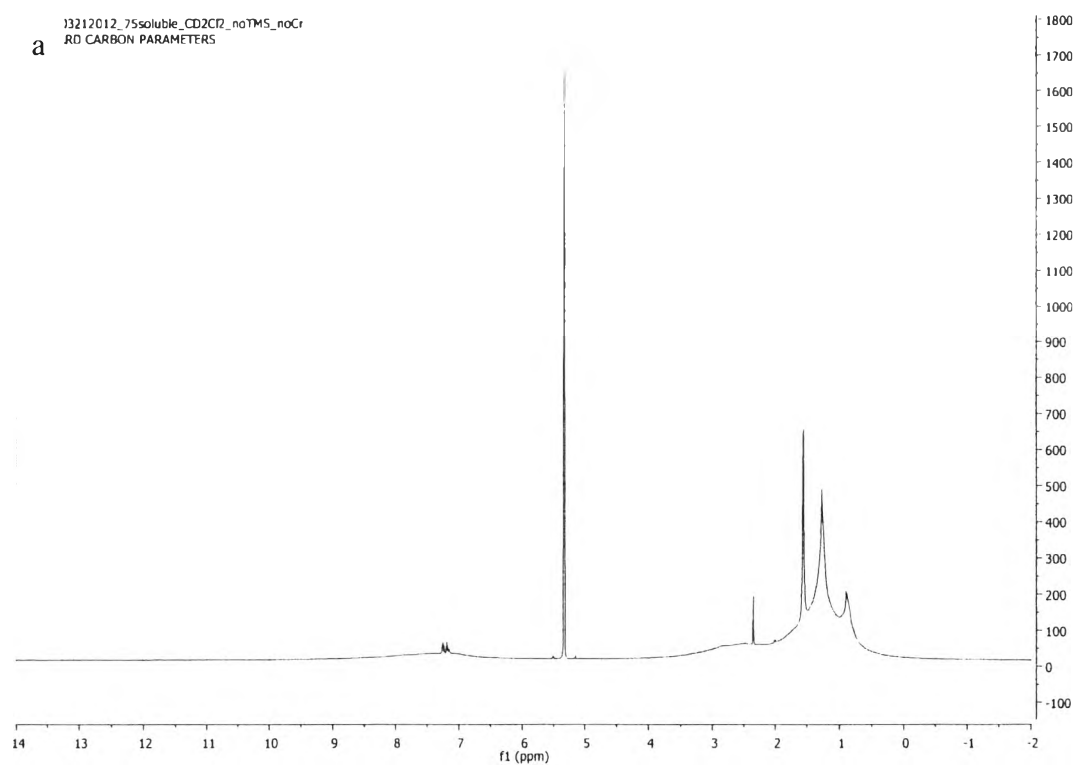


Figure F.2 NMR spectrum of 75 vol% heptane soluble asphaltenes (*Cut 8*) in deuterated chloroform (a) ^1H NMR (b) ^{13}C NMR.

The results of deuterated toluene and chloroform are similar that there is no asphaltene peak. Therefore, the better solvent for asphaltenes like deuterated methylene chloride was selected to increase the possibility of asphaltenes to soluble in solvent. The 75 vol% heptane asphaltenes (*Cut 8*) were dissolved in deuterated methylene chloride (1.32 wt%). Figure F.3 (a) shows both aliphatic and aromatic peaks of asphaltenes for ^1H NMR (0-4 ppm for aliphatic peak and 6-9 ppm for aromatic peak). But ^{13}C NMR still has only deuterated methylene chloride peak at 54 ppm (Figure F.3 (b)). It should have aromatic peak in the spectrum of ^{13}C NMR due to asphaltenes structure.



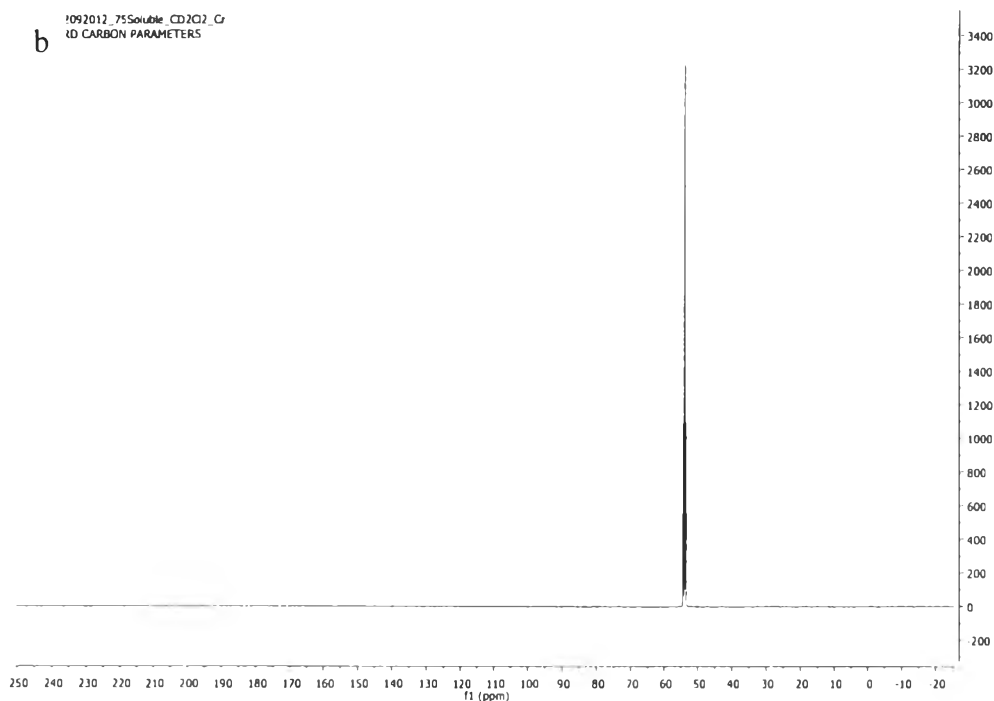


Figure F.3 NMR spectrum of 75 vol% heptane soluble asphaltenes (*Cut 8*) in deuterated methylene chloride (a) ^1H NMR (b) ^{13}C NMR at low concentration (1.32 wt%).

There is high amount of ^{12}C in asphaltene structure but lack of ^{13}C . To observe ^{13}C peak using ^{13}C NMR, 75 vol% heptane soluble asphaltenes (*Cut 8*) were dissolved in deuterated methylene chloride at very high concentration of asphaltenes (10 wt%). The ^{13}C NMR spectrum is shown in Figure F.4. ^{13}C NMR shows both aliphatic and aromatic peaks of asphaltenes at 10-60 ppm and 110-160 ppm, respectively.

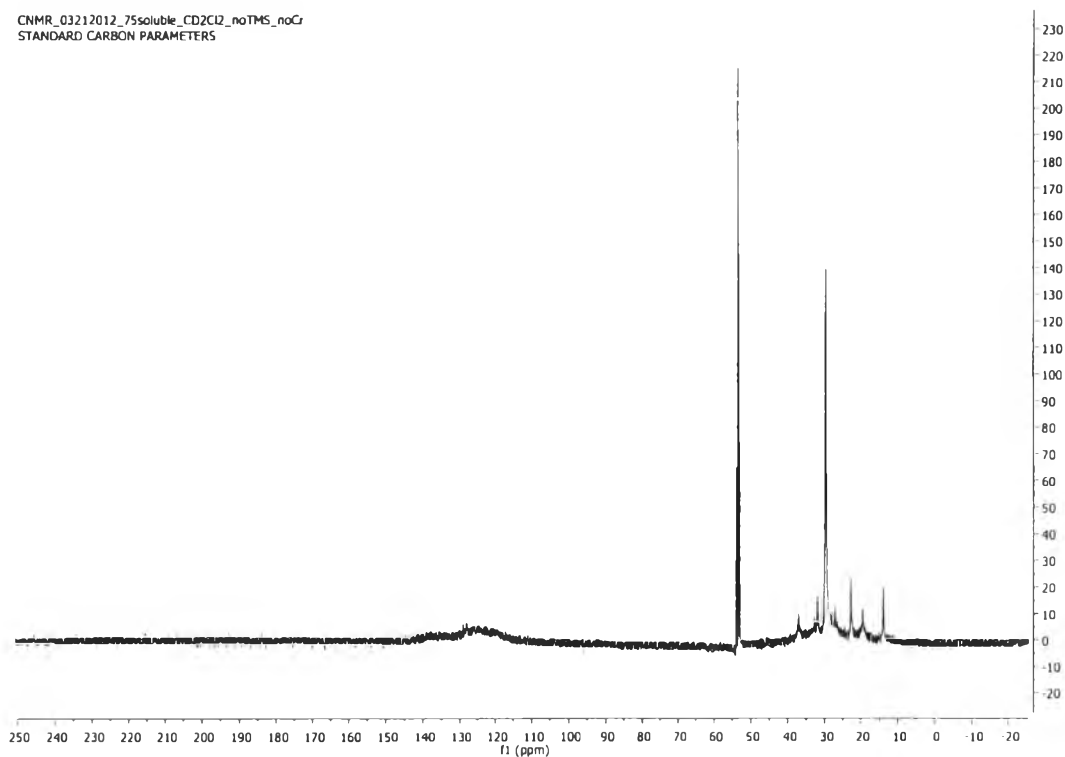


Figure F.4 ^{13}C NMR spectrum of 75 vol% heptane soluble asphaltenes (*Cut 8*) in deuterated methylene chloride at high concentration (10 wt%).

Therefore, deuterated methylene chloride was selected as solvent. The low concentration of asphaltenes was used for ^1H NMR because asphaltenes contain a lot of hydrogen in asphaltenes structure. For ^{13}C NMR, the high concentration should be used due to lack of ^{13}C in asphaltenes structure.

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