CORRELATION DEVELOPMENT OF WAX DEPOSITION PREDICTION FROM FANG OIL FIELD

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ในอุตสาหกรรมปิโตรเลียมการสะสมตัวของไขน้ำมัน เป็นความท้าทายทางการผลิตที่สำคัญ ซึ่งเกิดขึ้นในหลายๆ ตำแหน่งในห่วงโซ่การผลิตปิโตรเลียมรวมถึงในท่อขนส่ง สารเฮกเซนซึ่งเป็นสารยับยั้งทางเคมี เป็นวิธีการยับยั้งการสะสมไขน้ำมัน โดยจะถูกใช้เพื่อป้องกันปัญหาการสะสมของไขมันจากน้ำมัน ที่แหล่งฝาง โดยการวัดหาค่าจุดเท การวัดค่าอุณหภูมิที่ปรากฏไขและ การวัดค่าน้ำหนักของไขที่สะสมตัว การเพิ่มขึ้นของค่าอัตราเฉือน จะสามารถลดค่าดับบิวเอทีและปริมาณไขน้ำมันที่สะสม จะเพิ่มขึ้นเมื่ออุณหภูมิของน้ำมันดิบฝางมีค่าลดลง นอกจากนี้ ยังได้ดำเนินการทำการวิเคราะห์การถดถอยแบบไม่เป็นเส้นตรง ของน้ำมันที่แหล่งฝางที่มีและไม่มีสารเฮกเซน ในสภาวะต่างๆ ในการทดสอบทั้งสามแบบด้วยสมการ การวิเคราะห์ทาง รีเกรสชั่น สามารถทำนายค่าดับบิวเอที ที่อัตราเฉียนค่าต่างๆ และปริมาณไขน้ำมันที่สะสมที่อุณหภูมิต่างๆ ในน้ำมันจากแหล่งฝาง ได้มีการศึกษาผลของค่าตัวแปร เช่น ความเข้มข้นของสารเฮกเซน อุณหภูมิ อัตราเฉือนที่มีต่อจุดเท ค่าอุณหภูมิที่ปรากฏไข และการสะสมตัวของไขน้ำมัน ค่าอุณหภูมิที่ปรากฏไข และการสะสมตัวของไขน้ำมัน ความเข้มข้นของสารเฮกเซนที่เพิ่มขึ้นจะสามารถลดค่าจุดเท ยิ่งใช้ความเข้มข้นของเฮกเซนสูงขึ้น ค่าดับบิวเอทีจะลดลงได้มากขึ้น ทั้งความเข้มข้นของเฮกเซนและอุณหภูมิ ที่สูงขึ้น จะทำให้ ปริมาณไขน้ำมันสะสมลดน้อยลงดังกล่าว สามารถทำนายโดยใช้สมการสหสัมพันธ์ซึ่งดัดแปลงจากสมการของ โรนิงเซน มีการเปรียบเทียบค่าความแตกต่างในรูปของเปอร์เซ็นต์ระหว่างค่าที่ได้จากการทดลองและค่าที่ได้จากสมการ เปอร์เซนต์ความเข้มข้นของเฮกเซนและอัตราเฉือนที่เพิ่มขึ้น ทำให้ค่าดับบิวเอที ที่คำนวณได้ลดลงและสมการที่เลือกมาสามารถประยุกต์ใช้ได้ทั้งค่าความเข้มข้นและอัตราเฉือนความเข้มข้นของเฮกเซน ที่เพิ่มขึ้นและอุณหภูมิที่ลดลง จะช่วยลดปริมาณของไขน้ำมันสะสมโดยใช้สหสัมพันธ์ที่ดัดแปลงของรอนนิงเซ่น และสมการนี้สามารถใช้ได้ดีทั้ง ความเข้มข้นของสารและอุณหภูมิ ค่าต่างๆ

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In petroleum industry, wax deposition is a critical operational challenge. It occurs in various locations in the petroleum production chain including well tubing. Hexane, chemical inhibitor, is used to prevent the wax problem of oil from Fang oilfield by the measurement of pour point, wax appearance temperature (WAT) and wax deposition. An increasing shear rate can reduce WAT and the deposited amount increases with lowering the temperature in Fang crude oil. Moreover, the non-linear regression analysis of Fang crude oil with and without hexane in different conditions are done for all three tests. Those regression analysis equations can predict WAT at different shear rates and wax deposited weight amount at different temperature of crude oil in Fang. The effects of parameters such as hexane concentration, temperature and shear rate on pour point, WAT and wax deposition are studied. The higher hexane concentration can reduce pour point. The more hexane concentration was used, the higher WAT reduction was observed. Both higher hexane concentration and temperature give the less amount of wax deposit. The calculated WAT is attained from intersection point between equations and modified Ronningsen’s correlation is used for wax deposition prediction. The percent difference between the experimental and calculated results are compared. Both increasing hexane concentration percent and shear rate make calculated WAT decrease and the selected equations can be applied in both concentration and shear rate perceptive. Increasing hexane concentration and decreasing temperature can lower wax deposition amount in Modified Ronningsen’s correlation and this correlation works well with both different solvent concentrations and temperatures.

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LIST OF ABBREVIATIONS

AAD	average absolute deviation
ASTM D 5853-11	standard test method for pour point of crude oils
Avg.	average
CALC.	calculate
CONC.	concentration
deg	degree
diff	difference
Eq.	equation
EVA	ethylene-vinyl acetate
Exp.	experiment
PNA	paraffinic (p), naphthenic (n) and aromatic (a) fractions
PPD	pour point depressant
SARA	saturates, aromatics, resins, and asphaltenes
TEMP.	temperature
WAT	wax appearance temperature
WPC	the wax precipitation curve

NOMENCLATURE

A_w	wax deposition area (m^2)
c_1 & c_2	coefficients based on shear rate (6,12 & 24 per second) and chemical concentration percent (0%, 5%, 10%, 15% and 20%) in high temperature region of lab results
c_3 & c_4	coefficients based on shear rate (6,12 & 24 per second) and chemical concentration percent (0%, 5%, 10%, 15% and 20%) in low temperature region of lab results
$\frac{dM_w}{dt}$	wax deposited rate (kg/s)
D_B	Brownian diffusion coefficient (m^2/s)
$\frac{dC}{dr}$	concentration gradient over the pipe radial coordinate (1/m)
$\frac{dC}{dT}$	solubility coefficient of the wax crystal in the oil phase (1/ºC)
D_s	shear dispersion coefficient (m^2/s)
$\frac{dT}{dr}$	radial temperature gradient of the wall (ºC/m)
D_w	wax diffusion coefficient in the oil phase (m^2/s)
K_P	power-law consistency index
M_B	Brownian diffusion deposited wax mass (kg)
μ_r	relative viscosity which is apparent viscosity μ_r to continuous phase viscosity μ_c ratio
ρ_w	solid wax density (kg/m^3)
φ_w	volume fraction of wax out of the solution at the pipe wall.
μ	dynamic viscosity (cP)
μ	oil viscosity (Ns/m^2)
A and B	the system and shear rate dependent constants
a	Brownian particle diameter (m)
a	particle diameter (m)
A, B, C and D	coefficients of the correlation
a, b, c and d	multiple regression analysis constants

a_1, a_2, a_3 and a_4	coefficients of the correlation
CALC. wax wt.	calculated wax weight (g)
CONC.%	hexane concentrations at 5%,10%,15% and 20%
d	diameter of wax particle (m)
g	acceleration due to gravity (m/s^2)
k	constant depends on oil content and type of emulsion
K and n	constant
M	oil solvent molecular weight (g/mol)
n	power-law index
N	Avogadro’s number (1/mol)
R	gas constant (Jmol^{-1}K^{-1})
T	temperature (^{o}C)
T_a	absolute temperature (K)
U	settling velocity (m/s)
V	wax molar volume (cc/g mole)
V	dispersed phase volume
γ	shear rate (1/s)
ΔP	density difference between the oil and settling wax (kg/m^3)
μ	apparent viscosity
$\mu_{\text{High TEMP. Zone}}$	calculated viscosity in high temperature region (cp)
$\mu_{\text{Low TEMP. Zone}}$	calculated viscosity in low temperature region (cp)
ξ	association parameter representing the effective molecular weight of the solvent with respect to molecular diffusion
τ	shear stress (N/m^2)
ϕ	oil content

CHAPTER 1

INTRODUCTION

1.1 Overview of Wax Deposition

Wax deposition is a crucial operational challenge in industry of petroleum. It occurs in various locations in the petroleum production chain including well tubing. At high temperature, the waxy components, n-paraffins, dissolve in the crude oil but reaching the temperature below the WAT, waxy component precipitate into solids. This phenomenon causes high-pressure drop-in oil transportation causing blocking the inner wall of the pipe surface, the pipelines, and the process equipment. Wax deposition related operation problems are serious issues and therefore it is important to have a precise conception of wax precipitation/deposition’s physics and chemistry in order to evaluate mitigation and prevention plan with the economic perceptive (Huang et al., 2016).

1.2 Constituents of Hydrocarbons and Its Classifications

They are separated into aromatic and aliphatic with other organic compounds and nonhydrocarbon (Mc Cain Jr, 1990):

Aliphatic

Aliphatic is divided into following:

- alkanes,
- alkenes,
- alkynes,
- cyclic analogs.

Alkanes: The general formula is $C_n\ H_{2n+2}$ and called by a prefix which denotes the number of carbon atoms and the suffix -ane combination. The carbon atoms are attached to as many hydrogen atoms as possible are called paraffin hydrocarbons. The different carbon configurations are known as structural isomers or isomers. While the continuous chain hydrocarbons have prefix n-, the branched chain have prefix iso-. The prefix neo- denotes three methyl groups on a carbon atom at the end of a chain. An alkane with one hydrogen atom missing is an alkyl group named by using the prefixes corresponding to the carbon atom number.

Alkenes: Also called unsaturated hydrocarbons or olefins. The general formula is $C_n\ H_{2n}$. The feature is the carbon double bond which is a four-electron bond sharing two electrons from each of two carbon atoms. The suffix is -ene.

Alkynes: The carbon-carbon triple bond structure with the formula of $C_n\ H_{2n-2}$.

Cyclic: The carbon atoms are arranged in rings and called cyclic compounds.

Aromatics

Aromatics includes benzene and benzene resembling compounds. It formed with benzene as the building block.

Petroleum Crude Oil Classification

Crude oil can be classified based on physical properties or constituent chemical molecule structures (Mc Cain Jr, 1990).

Physical Classification

Through measurement, petroleum liquid commercial value can be estimated;

- specific gravity,
- cloud point,
- pour point,
- sulfur content and
- gasoline and kerosene content.

Among these tests, cloud point and pour point can qualitatively be measurable the liquid paraffin content.

Chemical Classifications

This classification is related to the molecular structure in the oil. Six carbon atoms and less are mostly paraffins. After most of the light molecules are removed, this analysis has made. Terms such as paraffinic, naphthenic, naphthenic-aromatic and aromatic-asphaltic are used in the several classification methods. They are related to the molecular structure of the chemical species most prominent inside oil. The classification of crude oil is little important in its production except that paraffinic crudes can precipitate wax and plug the production string.

Chemical Classification of Petroleum Fluids

Components of the high-molecular-weight deposition depends on the reservoir fluid which is a mixture of multicomponent. The chemical constituents separated as either the C6- which is light end or the C6+ fraction which is heavy end.

Classification of Petroleum Constituents

Describe components as:

- paraffinic,

- naphthenic, or

- aromatic fractions.

They all together was known as PNA.

The Paraffins

N-alkanes and i-alkanes with single bond hydrocarbon segment chains. The most solid-wax deposits was caused by high molecular weight paraffins.

Naphthenes

Naphthenes includes the cycloalkanes which contains one or more cyclic structures. Single bonds joined the cyclic structure element. Large part of microcrystalline wax is made up by naphthene.

Aromatics

Consist all compounds with ring structure, six identical, of one or more.

Resins and Asphaltenes

Although some may contain only naphthenic rings, they are a subclass of the aromatics and consisting large molecules mostly carbon and hydrogen with one to three sulfur, oxygen, or nitrogen atoms per molecule. Composed of rings, aromatics majority with ten to three or more rings.

Petroleum constituents' SARA Analysis

The components heavy fraction components can be separated into four groups:

- Saturates- all components of hydrocarbon with atoms of carbon in single bond.
- Aromatics- benzene and all the derivatives at least one benzene ring.
- Resins- included aromatic, rings of naphthenic and heteroatoms as a group of highly polar end. Sticky solids or heavy liquids as a pure form.
- Asphaltenes- group of most high polar components such as condensed aromatic, rings of naphthenic and heteroatoms and asphaltenes pure are black and powders of nonvolatile.

SARA analysis was called to each group fraction determination (Lake & Fanchi, 2006).

1.3 Study Background

Fang oil field is facing with wax deposition problem. It is a common and challenging production problems in many oil fields. In this study, used oil samples from Mae Soon reservoir shall be evaluated using hexane as a chemical inhibitor. Correlations and regression analysis shall perform base on test results.

1.4 Objective of the Study

1. To measure the WAT, pour-point temperature and wax deposited amount from Mae Soon conditions.
2. To determine hexane concentrations for wax deposition prevention by measuring the WAT, pour point temperature and wax deposited amount in Mae Soon Reservoir at Fang oilfield.
3. To develop the correlation for wax deposition prediction.

The scope of this work is to understand wax deposition problem in production and how to handle it. This will achieve by analyzing results from pour point test, wax appearance temperature (WAT) test and wax deposition amount test. The hexane concentration effectiveness, regression analysis and correlation works can compare with previous studies to mitigate wax deposition problem in the Fang oil field. This experimental result shall be useful for wax deposition related future studies

Chapter one has described the overview of wax deposition, crude oil classifications, background, objective and scope of this research. Chapter two will describe the physics and chemistry of wax deposition, its problems, prevention and remediation in the past as literature review. Chapter three includes methodology for tests and correlations. Chapter four will present results from different approaches. Conclusions and recommendation will be in Chapter five.

CHAPTER 2

LITERATURE REVIEW

2.1 Wax Deposition Behavior Mechanism

Wax component precipitation occurs when the original reservoir equilibrium conditions are changed. Wax component precipitates as the wax solubility has reduced. Not all wax precipitation become deposition. The particles detach from the fluid and form deposits when crystals of wax become large particles. The vertical solid and liquid phase boundary indicates wax precipitation is strongly dependent on temperature and merely dependent on pressure.

The most common wax deposition is caused by temperature reduction. When the temperature decreases, wax solubility in hydrocarbon fluids decreases.

Cooling can be caused by;

- expansion of oil and gas along the chain;
- liberated solution gas;
- fluid heat radiation to the surrounding when its travel to the wellbore;
- when the fluid was transferred through the surface facilities at low temperature; and
- other fluids or water injection at below the reservoir temperature.

The cloud-point increases when the solution gas liberated from the crude oil below the bubble point (Lake & Fanchi, 2006).

Wax Deposition Leading Factors

Leading factors for wax deposition are:

a) Temperature: Temperature have direct relationship with the paraffin solubility. The temperature gradient of the bulk oil and the cold pipe wall leads to wax deposition.
b) Crude oil composition: Components are in equilibrium, aromatics and light end saturates act as solvents for high molecular weight saturates and heavy ends, at initial but production changes and resulting the solubility of the paraffin waxes decreases.
c) Pressure: The reservoir pressure decline causes the solute solvent ratio increase. The wax appearance temperature (WAT) increases with pressure rises above the bubble-point but reduces with rising pressure under the bubble-point.

d) Flow rate: Mixing occurs in turbulent flow regime. Mixing does not occur in laminar flow regime. Laboratory investigations reveal deposition is affected by flow of laminar rather than turbulent.

e) Gas/oil ratio: Beyond the bubble point, gas in the solution helps to keep wax. Wax appearance temperature (WAT) is high with low GOR (Gas-Oil ratio) .

Wax Deposition Effects

Wax deposition has three effects (Kelland, 2014):

a) Gradual pipe restrictions – reduced flow resulting from both pipe diameter reduction and roughness of the pipe wall.

b) Increasing fluid viscosity - leads to the line pressure loss and gelling fluid.

c) Formation of wax gel - the line cannot be operated when the yield stress of the gel, occur during the shutdown of the pipeline, is higher than the maximum pressure of the pipeline.

Deposited Wax Detection

Pressure echo techniques, pigging and the ‘‘take-out’’, pressure drop, and heat transfer methods can be used to detect wax deposition. Also different methods were only tested with small-scale designs (Aiyejina et al., 2011).

2.2 Characteristics of Petroleum Waxes

Wax precipitation is mainly temperature-dependent rather than pressure-dependent (Huang et al., 2016). Wax is formed from alkanes and cycloalkanes with carbon number ranging between 18 to 65.

Two petroleum wax can be classified into two classes including:

❖ Paraffin waxes which are large flat plate normal alkanes that crystallize in the structure of macrocrystalline

❖ Microcrystalline waxes which are alkanes and cycloalkanes that crystallize in the structure of small needle

Parraffin waxes have molecular weight of 350 to 600, whereas microcrystalline waxes have 300 to 2,500. Crystal structures of deposited wax will be deformed because of wax precipitation complex in production environment with the mixing of wax types (Lake & Fanchi, 2006).

Formation of solid wax includes two important stages:

❖ Nucleation

❖ Growth of crystal

Upon reaching critical size and becoming stable, wax clusters continue to attach and detach from nuclei, heterogeneous or homogeneous nucleation, when the temperature falls below the WAT. After a plate-like or lamellar molecule are formed, the processes of crystal-growth occur.

Normal alkanes can separate into:

- hexagonal which has odd (11 to 43) or even (22 to 42) carbon numbers, and has high degree of molecular-rotational freedom and both plastic and translucent characteristics,
- orthorhombic which has 43 or greater carbon numbers
- triclinic which has from 12 to 20 carbon numbers, and
- monoclinic which are considered by even number of alkanes from the hexagonal or orthorhombic of the cooling (Lake & Fanchi, 2006).

2.3 Wax Thermodynamic Parameters

Various experimental techniques are used to characterize two important thermodynamic parameters including:

- wax-appearance temperature (WAT) and
- wax precipitation curve (WPC).

WAT gives wax deposition location and WPC provides wax solubility limit. Both of them are input parameters required for modeling of wax deposition and generally applied as wax thermodynamic model benchmarking references (Huang et al., 2016).

Wax appearance temperature (WAT) determination

WAT normally refers to noticeable wax precipitation temperature and apply concept of oil physical properties changes because of wax-crystal formation. Crude oil acts as a Newtonian fluid and oil viscosity as a function of temperature can be showed by Arrhenius-type equation with temperature above WAT. Wax precipitation and suspended wax in liquid are downed and crude oil flow properties are changed because of the suspended particles. This method can be applied as the slope change of viscosity as a function of temperature and an extrapolation from the Newtonian region can be found the WAT. This can be achieved by an exponential fitting of μ as a function T or $\ln(\mu)$ as a function of 1/T as shown in Figure 2.1 (Huang et al., 2016) (Roenningsen et al., 1991).

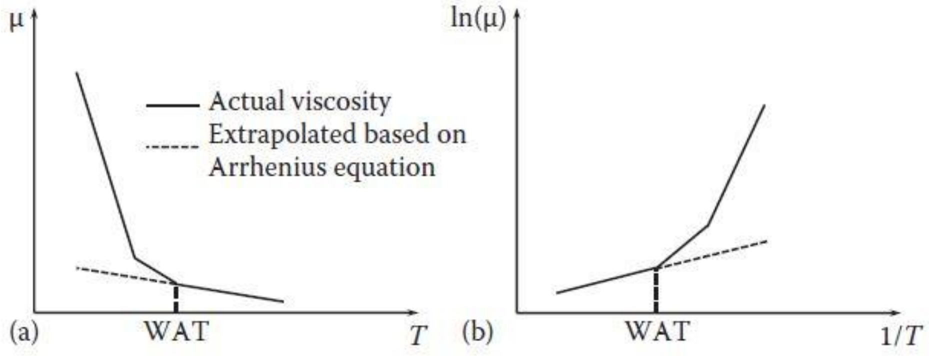


Figure 2. 1 (a) Crude oil viscosity as a function of temperature with the extrapolation from the Newtonian region of the curve. (b) ln(μ) as a function of 1/T with extrapolation from the Newtonian region

(Huang et al., 2016)

The Wax Precipitation Curve (WPC) Determination

Changes in physical properties caused by the wax precipitation are linked with wax amount. WPC gives precipitated amount of wax at temperatures below WAT as shown in Figure 2.2. The curve is obtained by measuring the solid cake weight precipitated at temperatures below the WAT after cooling the sample (Huang et al., 2016).

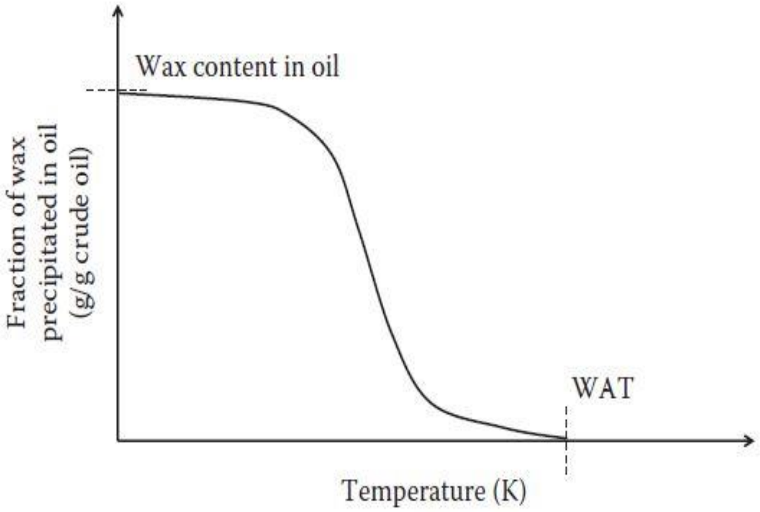


Figure 2. 2 the WPC of a crude oil example

(Huang et al., 2016)

2.4 Wax Deposition Mechanisms

Wax deposit formation mechanisms are as follows;

1. Molecular diffusion: waxy components dissolved molecules diffusion toward the pipe wall,
2. Shear dispersion: the waxy components precipitated particles dispersion toward the pipe wall,
3. Brownian diffusion: the precipitated particles diffusion by Brownian toward the wall
4. Gravity settling: the waxy precipitated particles settling toward the pipe bottom.

Dissolved wax molecules are subjected on deposition in the first mechanism and the precipitated suspended wax particles are the main reason for the rest mechanisms (Huang et al., 2016). The last three mechanisms are assumed as secondary mechanisms. The third mechanism is not likely to occur because the temperature of the wall temperature is less than that of oil. The final mechanism is also not important since deposits are not generally thicker at the pipe bottom and also particles were dispersed in pipelines by forces (Theyab, 2018). The deposition rate of wax does not increase with the shear rate, meaning that the second mechanism has less chance of occuring (Huang et al., 2016).

The region of rotating fluid applies a drag force on nearby particles. Therefore, each of them interacts with nearby particles when moving in a field. Dispersion of the shear can be evaluated using equation (2.1) (Burger et al., 1981):

$$D_s = \frac{\gamma d^2 \varphi_w}{10} \qquad (2.1)$$

where,

D_s = shear dispersion coefficient (m^2/s)

γ = shear rate at the pipe wall (1/s),

d = diameter of wax particle (m),

φ_w = volume fraction of wax out of the solution at the pipe wall.

Brownian movements happened when oil molecules and wax crystals collies and it can favor the wax particles movement when the gradient of concentrations exist. Brownian diffusion can be modelled by Fick’s law as shown in equation (2.2) (Theyab, 2018).

$$\frac{dM_B}{dt} = \rho_w D_B A_w \frac{dC}{dr} \tag{2.2}$$

where,

M_B = Brownian diffusion deposited wax mass (kg),

ρ_w = solid wax density (kg/m^3),

D_B = Brownian diffusion coefficient (m^2/s),

A_w = wax deposition area (m^2),

$\frac{dC}{dr}$ = concentration gradient over the pipe radial coordinate ($\frac{1}{m}$).

D_B are derived by the equation (2.3)

$$D_B = \frac{R\,T_a}{6\,\pi\,\mu\,a\,N} \tag{2.3}$$

where,

R = gas constant (Jmol^{-1}K^{-1}),

T_a = absolute temperature (K),

μ = oil viscosity (Ns/m^2),

a = Brownian particle diameter (m),

N = Avogadro’s number (1/mol).

Crystals of wax precipitation are heavier than near and settle in a field of gravity and can be deposited to the bottom. The settling velocity U can be found by the modified Stokes’ law,

$$U = \left[\frac{g\,\Delta P\,a^{(1+n)}}{18\,K_P}\right]^{1/n} \tag{2.4}$$

where,

U = settling velocity (m/s),

ΔP = density difference between the oil and settling wax (kg/m^3),

n = power-law index,

g = acceleration due to gravity (m/s^2) and

K_p = power-law consistency index.

The shear rate and shear stress correlation are:

τ = $Kp\ \gamma^n$ (2.5)

where,

τ = shear stress (N/m²) and

γ = shear rate (1/s).

Molecular Diffusion Wax Deposition Mechanism

There is a temperature gradient when the oil is being cooled. The molecular diffusion will transport dissolved wax toward the wall which lead to a concentration gradient during the temperature profile difference. Wax-gel formation and the deposited wax gel aging are the two stages in this mechanism.

The mechanism modelled by Fick’s law (Theyab, 2018):

$$\frac{dM_w}{dt} = \rho_w\ D_w\ A_w\ \frac{dC}{dr} = \rho_w\ D_w\ A_w\ \frac{dC}{dT}\frac{dT}{dr} \qquad (2.6)$$

where,

$\frac{dM_w}{dt}$ = wax deposited rate (kg/s),

D_w = wax diffusion coefficient in the oil phase (m²/s),

$\frac{dC}{dT}$ = solubility coefficient of the wax crystal in the oil phase (1/ºC),

$\frac{dT}{dr}$ = radial temperature gradient of the wall (ºC/m).

The diffusion coefficient can be showed:

$$D_w = 7.4*10^{-9}\ \frac{T_a\ (\xi M)^{0.5}}{\mu V^{0.6}} \qquad (2.7)$$

where,

M = oil solvent molecular weight (g/mol),

V = wax molar volume (cc/g mole),

μ = dynamic viscosity (cP) and

ξ = association parameter representing the effective molecular weight of the solvent with respect to molecular diffusion

The following four steps involved in this mechanism:

Step 1: Dissolved molecules of wax precipitation

Once the temperature reaches below the WAT, the waxy components start to precipitate and form crystals.

Step 2: Dissolved waxy components radial concentration gradient generation

A wax component radial concentration difference, obtained from wax components precipitation difference due to different temperature during normal cooling conditions, is generally greater at the wall than in the bulk. The components diffuse from a higher concentration bulk oil toward the wall, lower concentration place.

Step 3: Waxy Component deposition on the existing deposit surface

The precipitated components on the wall surface contributes to the wax deposit formation. The components diffusion toward the deposit occurring continue and resulting deposit buildup.

Step 4: Waxy Components internal diffusion in the deposit

Waxy component continues diffuse into the deposit and resulting the wax fraction increase known as deposit aging (Huang et al., 2016).

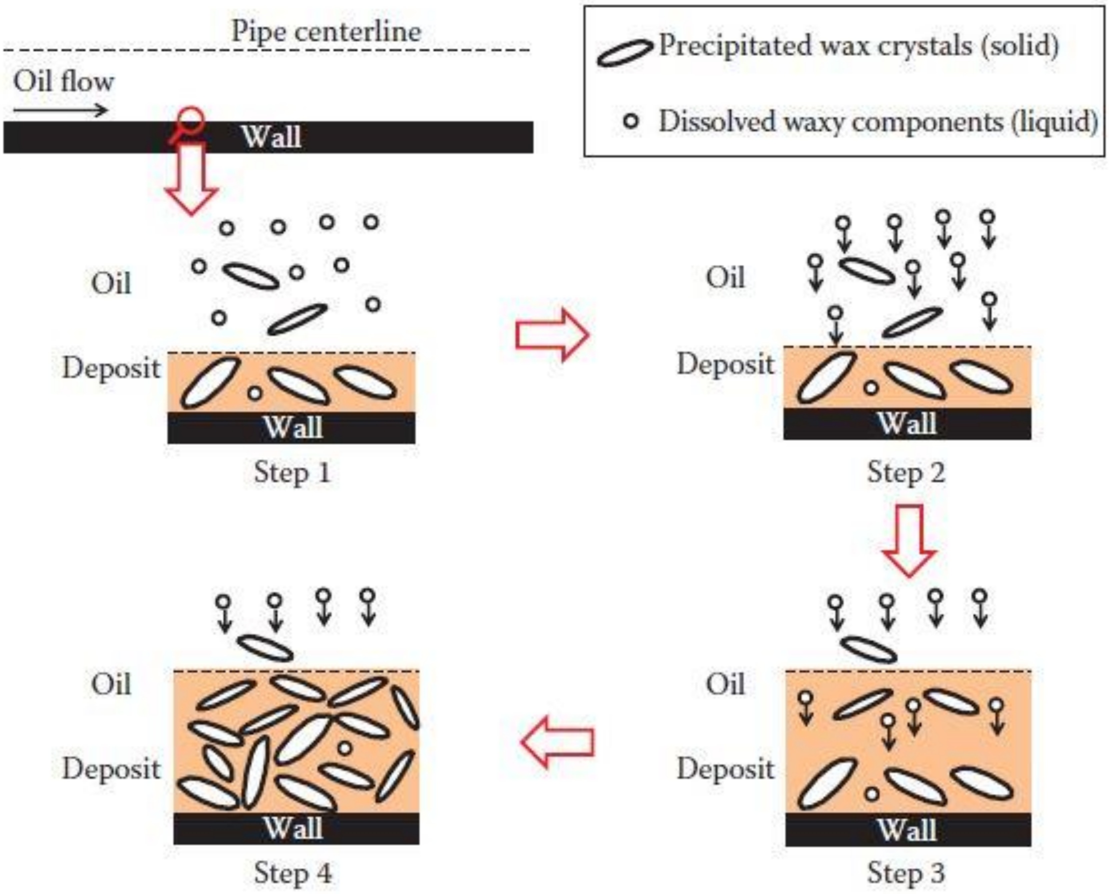


Figure 2. 3 Molecular diffusion wax deposition mechanism schematic

(Huang et al., 2016)

2.5 Wax Deposition Experiments

The modelling of wax deposition measurement predicts deposition severity and possibility to achieve effective prevention and remediation methods. Lab-scale experiments are conducted in a controlled environment. As the flow field in a flow-loop is like in field and the scale-up more similarity than others, it was assumed to be the best. Well-designed tests serve not only benchmarking model base but also give information for theoretical research and development. Two parameters measured are:

- the deposit thickness and
- the deposit waxy components composition (Huang et al., 2016).

Cold-Finger Wax Deposition Apparatus

Cold-finger testing apparatus is less costly and requires smaller oil volume and is used for the screening chemicals as shown in Figure 2.4.

The cell is equipped with a stirrer incooperated with a probe. Cold fluids are circulating inside the probe which is put inside the container of oil, which has a heating or cooling circulation system, so that deposit will form on its outer surface during test. Various cells of deposition can be placed inside the container for effectiveness test (Huang et al., 2016).

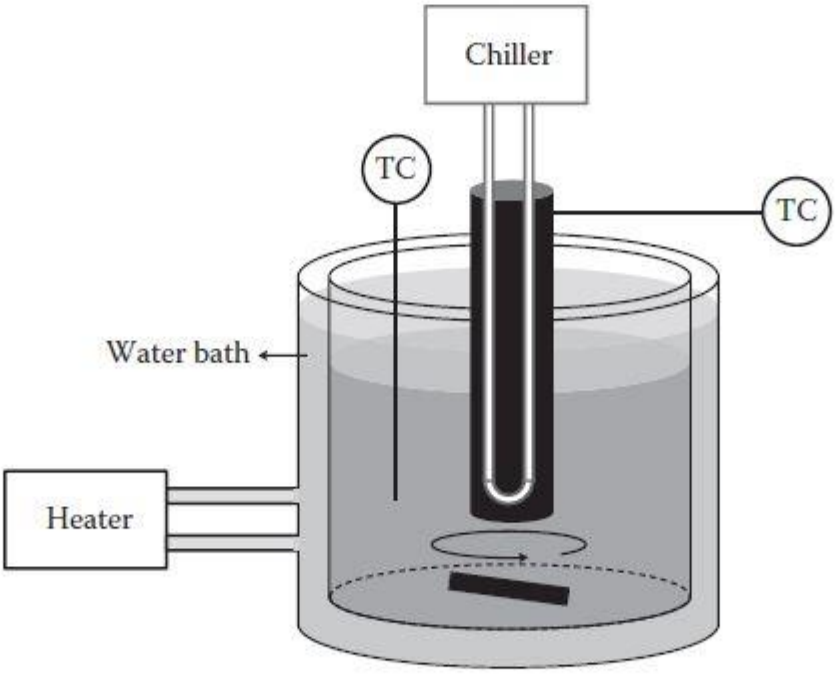


Figure 2. 4 The cold-finger wax deposition apparatus

(Huang et al., 2016)

2.6 Wax Deposits Removal and Prevention Mechanisms

Success rate of remediation techniques is depended on wax composition and crude oil type (Thota & Onyeanuna, 2016). Removal methods are (Clegg, 2007):

- mechanical,
- solvents and
- thermal.

Comingle production, pipe insulating, shock chilling or cold flow, permanent magnets and electromagnets conditions, placing a heating element by various ways, microbial and ultrasonic treatments can manage gelling and deposits buildup (Kelland, 2014).

Mechanical Method for Wax Deposition Removal

Mechanical removal by means of scrapers and cutters, is done regularly to prevent wax deposit buildup as pipeline pigging is common in fuel industry. This is relatively simple but needs shutdown. Wax can cause perforation plugging whenever it is necessary to circulate scraped paraffin down the tubing and out of the casing. This method becomes costly when frequent cleanout is required. Attaching a scraper to the wireline is widely used in paraffin removal method (Allen, 1981) (Kelland, 2014).

Thermal Method for Wax Deposition Removal and Prevention

Electric and steam surface heating combine with insulation are methods of prevention. Hot oiling and hot watering are removal methods (Lake & Fanchi, 2006). The first one uses heat to melt and remove the wax dissolve and melt the paraffin and then circulated back. Oil is heated to a temperature greater than that of the formation. Hot watering or steam has been used to melt Paraffin or asphaltenes in the flowline, tubing, casing, wellbore or formation. Permeability damage can happened because of melted wax (Allen, 1981).

Chemical Method for Wax Deposition Removal and Prevention

The selection of a solvent for any application should be based on its cost effectiveness in dissolving a specific organic deposit. Soaking or surging of the solvent over a period will usually dissolve paraffin maximum per gallon of solvent. Application must be adapted to fit well conditions (Allen, 1981). Famous solvents are gas oil blending, substituted aromatics and hot water. A solvent is a composition of an aromatic and an alicyclic and/or aliphatic hydrocarbon. A surfactant may conation in solvents of wax. Hot solvents, aromatic solvents, a terpenoid proprietary blend, oxygenated and solvents based on hydrocarbon and kerosene will takeout deposits. Magnesium oxide dispersed powder served as the breaker for viscosity.

Crystals of wax were modified so that they cannot gather and deposit. Wax inhibitors are chemicals that can change WAT. Pour point affected on chemicals were called as a flow improver or PPDs. Both have the same mechanisms and chemical characteristics. They should be applied before the temperature reaches under WAT. Wax strength decides the concentration that should apply for both. The PPDs must be applied doubly with the increasing content of asphaltene. The first one cannot stop the deposition, but it can reduce frequency of wax removal and the second one can be used to stop wax gelling. Molecular crystallization will interfere or modify wax by co-crystallizing or interacting with the wax when another part can stop the continuous wax growth by wrapping new wax molecules attaching places

The main classes of wax inhibitors and PPDs can be summarized as follows:

- copolymers and ethylene polymers,
- miscellaneous branched polymers with long alkyl groups and
- comb polymers.

The wax adhesion is deceased by surfactants changing water wet or creating a weak layer on surface or adsorbing on particles and cannot move freely near. For inhibitory effect, continuously dosed is needed. Polymeric was mixed to increase effect of dispersants. Alkyl sulfonates, alkyl aryl sulfonates, fatty amine ethoxylates and other alkoxylated products are also used as wax dispersants (Kelland, 2014).

Deployment Techniques

The most common deployment method is injection at the wellhead. Chemicals can be injected downhole with a string of capillary and wax inhibitor dispersion (Kelland, 2014).

2.7 Viscosity of Crude Oil Correlation Works

By power-law relationship, the emulsion rheological behavior within the shear rate practical range can be described as follow:

$$\tau = K\,\gamma^{n} \tag{2.8}$$

where,

τ = shear stress,

γ = shear rate and

K and n = constants.

The apparent viscosity μ is defined:

$$\mu = \frac{\tau}{-\gamma} = K\,\gamma^{n-1} \tag{2.9}$$

For fluids with Newtonian behavior, n is equal to 1 and μ is equal to k. n ranges from 0 to 1 and the negative exponential value for fluids with mostly pseudoplastic behavior and n shifts near zero and n-1 value nears -1 for fluids with highly pseudoplastic. The correlation between the emulsion apparent viscosity and oil content is:

$$\mu_r = e^{k\,\phi} \tag{2.10}$$

where,

μ_r = relative viscosity which is apparent viscosity μ_r to continuous phase viscosity μ_c ratio,

ϕ = oil content and

k = constant depends on oil content and type of emulsion.

The emulsion viscosity links with the temperature so that ln μ versus 1/T plot gives a positive slope straight line and appears as an Arrhenius-type equation:

$$\mu = A\, e^{\frac{B}{T}} \qquad (2.11)$$

where,

A and B = the system and shear rate dependent constants

New equation modified from previous equations is applied to find the O/W emulsion viscosity with variable functions , shear rate, oil content and temperature (Al-Roomi et al., 2004):

$$\mu = a\, \gamma^{b} \exp\left(c\, \phi + \frac{d}{T}\right) \qquad (2.12)$$

where,

a, b, c and d = multiple regression analysis constants

Ronningsen (1995) has presented the viscosity of water and oil emulsions as a function of volume fraction of dispersed phase and temperature correlation:

$$\ln \mu_r = a_1 + a_2T + a_3V + a_4TV \qquad (2.13)$$

where

V = dispersed phase volume and

a_1, a_2, a_3 and a_4 = coefficients of the correlation.

Juntarasakul (2015) measured emulsion viscosity and evaluate emulsion stability of light oil from Fang Oilfield and developed correlations to predict oil and emulsion viscosity and stability.

The Newtonian region extrapolation in the temperature vs viscosity curve will give wax appearance temperature (WAT) (Huang et al., 2016).

2.8 Research Gap

Literature review suggested that only few solvents have been investigated on reduction of wax deposition at low concentration. It is needed to predict WAT (wax appearance temperature) by equations, to do wax deposited amount correlation, to compare effectiveness of chemicals in Fang's crude oil under different conditions.

CHAPTER 3

METHODOLOGY

3.1 Collecting Materials

Crude oil sample is obtained from Fang oil field of Northern Thailand and n-hexane is purchased from Sigma-Aldrich for this study.

3.2 Used Equipment and Test Procedure

Following tests will make to crude oil and Fang crude oil with different hexane concentrations to evaluate its effectiveness (Numura, 2005) (Dehaghani & Badizad, 2016) (Banerjee et al., 2017).

Pour Point Test
Wax Appearance Temperature (WAT) Test and
Wax Deposition Test

3.2.1 Pour Point Test

The lowest temperature where sample movement is found under conditions of the test.

Equipment

The temperature measurements are performed by Kimo thermocouple data loggers' model KTT 220 and thermocouples J-Type with ± 0.4°C accuracy and temperature ranging from 0 to 1,300°C.

Test Procedure

Crude oil pour point with inhibitors in concentration differences and without inhibitors is tested by following ASTM D 5853-11.

1. The test sample is heated at least 20°C above expected pour point with water bath.
2. The temperature is reduced in the multiple of 3°C and begins to examine the appearance of sample
3. The test tube is tilted just enough to ascertain every 1°C down whether there is movement of the sample in the test tube or not.

4. If the movement of the test sample is shown, the test tube is replaced immediately in the water bath and repeat a test for the flow at the next temperature, 3°C lower.
5. When the test sample shows no movement stops the test and the test tube is held in a horizontal position
6. Record that Temperature (Min, 2018).

That recorded temperature is the pour pint temperature.

3.2.2 Wax Appearance Temperature (WAT) Test

Equipment

Brookfield Viscometer model DV2TLV with spindle number 52Z (4.6 to 92130 cp) is used to perform the viscosity measurements. Julabo F26 model heating or cooling bath machine with ± 1 accuracy is utilized to control temperature and glycol is used as a heating and cooling media as shown in Figure 3.1.

Test Procedure

WAT is measured by using viscometer.

1. The crude oil is heated up to 80°C before the test and
2. The crude oil under various conditions, with inhibitors at different concentrations and without inhibitor, is transferred to the viscometer cup
3. The temperature is cooled down ranging from 80°C to 40°C with shear rate differences and constant cooling rate (Min, 2018).

Figure 3. 1 Viscometer with temperature-controlled bath

(Min, 2018)

3.2.3 Wax Deposition Test

Equipment

Rod, a 25 cm long of copper stick, is equipped at rubber cork together with thermocouple J-type. It is designed to evaluate the wax inhibitors performance as shown in Figure 3.2. Crude oil temperature is controlled by using temperature controller bath Julabo immersion circulator ME model heating or cooling bath machine with ± 0.01 accuracy. Water is used as a heating and cooling media to control temperature.

Test procedure

Wax deposition determination is conducted by cold finger technique.

1. The weight of the naked rod is measured.
2. Crude oil is melted down at 65°C before commencing wax deposition test with inhibitors in different concentrations and without inhibitors.
3. Transfer sample into test tube
4. Close with cork carrying cold finger and thermocouple.
5. Conducted for 3-hour period with different temperatures and concentrations to investigate the temperature gradient effect.
6. By the precision 4 digits weighing machine, wax deposits mass is measured.
7. The experiment is repeated for 3 times and gets the average result (Min, 2018).

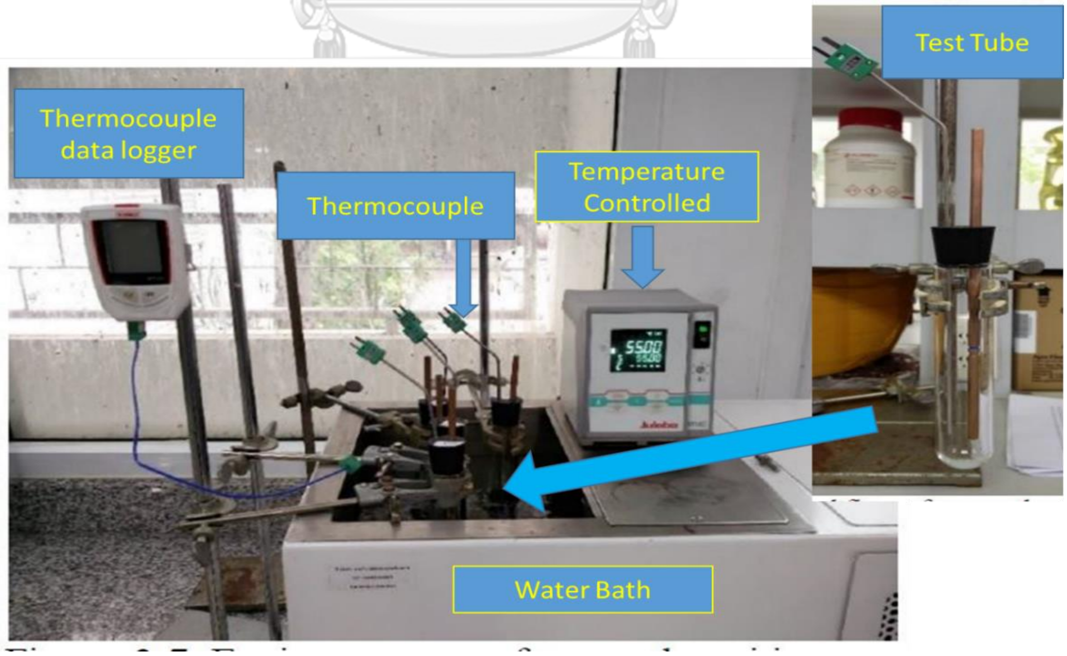


Figure 3. 2 Pour point and wax deposition test equipment setup

Modified from (Min, 2018)

3.3 Calculating Wax Appearance Temperature (WAT)

An Arrhenius-type equation got when plotting μ versus T (Roenningsen et al., 1991) (Al-Roomi et al., 2004),

$$\mu_{\text{High TEMP. Zone}} = c_1 * e^{c_2 * T} \qquad (3.1)$$

$$\mu_{\text{Low TEMP. Zone}} = c_3 * e^{c_4 * T} \qquad (3.2)$$

where,

$\mu_{\text{High TEMP. Zone}}$ = calculated viscosity in high temperature region (cp)

$\mu_{\text{Low TEMP. Zone}}$ = calculated viscosity in low temperature region (cp)

c_1 & c_2 = coefficients based on shear rate (6,12 and 24 per second) and chemical concentration percent (0%, 5%, 10%, 15% and 20%) in high temperature region of lab results

c_3 & c_4 = coefficients based on shear rate (6,12 and 24 per second) and chemical concentration percent (0%, 5%, 10%, 15% and 20%) in low temperature region of lab results

T = temperature (°C)

To find coefficients in each condition, results from lab tests are plot. First, coefficients c_1 and c_2 are obtained from high temperature region. Second, coefficients c_3 and c_4 are obtained from low temperature region in the viscosity vs temperature lab result curve. Those coefficient values are shown in Appendix E. Third, calculate viscosities at different temperature using those coefficients in each condition, different shear rates and chemical concentrations, by the high temperature region equation, the shear rate independent viscosities, and low temperature region equation, the shear rate dependent viscosities. Finally, wax appearance temperature (wat) will get from intersection point of previous two equations in viscosity at variance with temperature curve.

3.4 Predicting Wax Deposition Amount

Correlations used for predicting wax deposition amount are modified from (Ronningsen, 1995). He correlated viscosity with temperature and water content. This correlation adopted the concept and create correlation between deposited wax amount with chemical concentration and temperature.

Following correlation is developed from (Ronningsen, 1995):

ln (CALC. wax wt.) = A + B*CONC.%+ C*T + D*CONC.%*T (3.3)

where,

CALC. wax wt. = calculated wax weight (g)
CONC.% = hexane concentrations at 5%,10%,15% and 20%
T = temperature (°C)
A, B, C and D = coefficients of the correlation

Multiple regression analysis is done in average deposited wax weight tests to get the correlation coefficients.

Table 3. 1 Coefficients for modified Ronningsen's correlation

Modified Ronningsen's correlation	
A	2.907630
B	-0.115418
C	-0.061315
D	0.000921

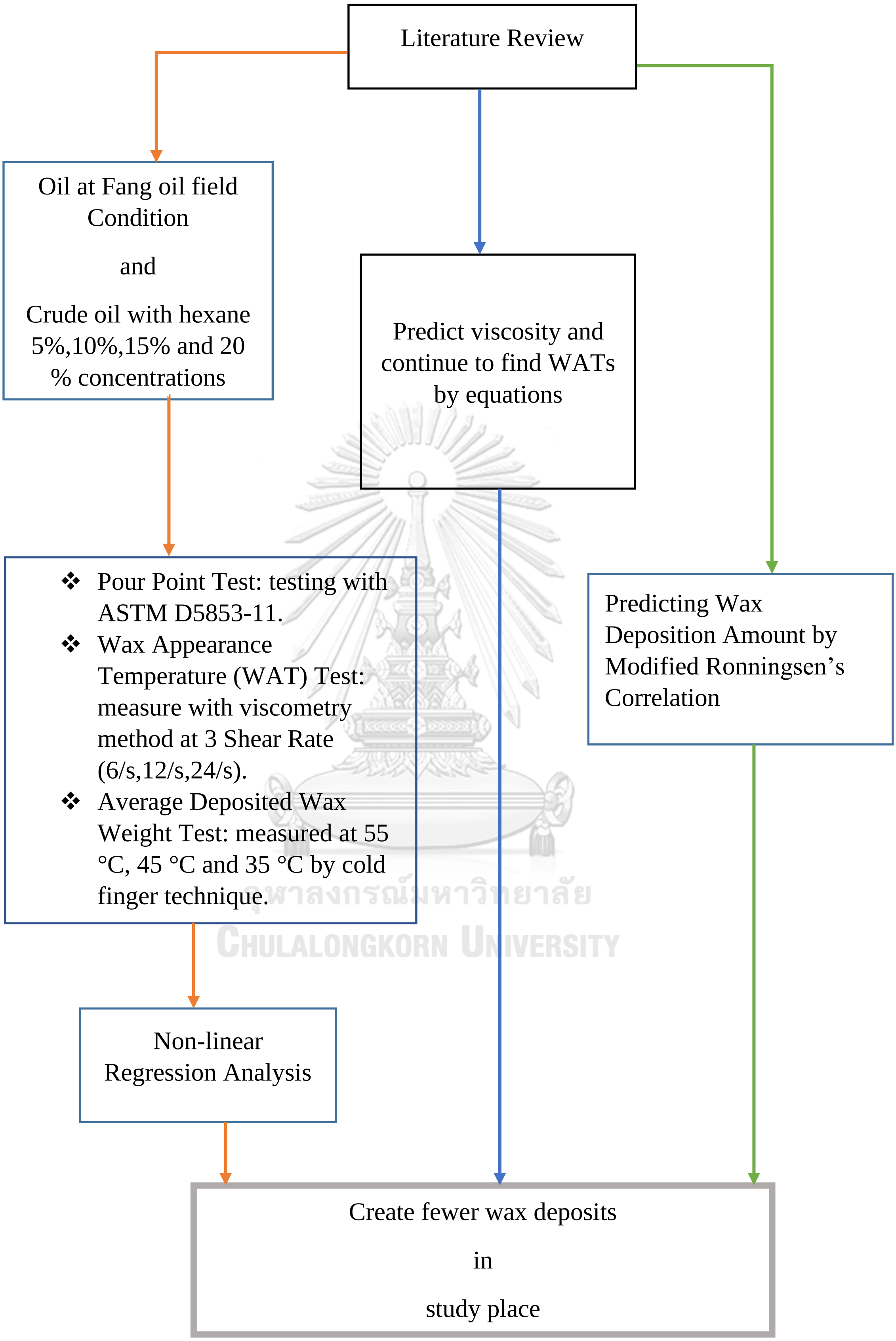


Figure 3. 3 Flow chart of study

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Fang Crude Oil Characteristics

At room temperature, crude oil from Fang oil field is solid state condition with higher wax content. After testing with ASTM D 5853-11 method, its pour point is 36°C. The wax appearance temperatures (WATs) of crude oil are 44.5°C in shear rate 6/s, 44.5°C in shear rate 12/s and 42.5°C in shear rate 24/s tested using viscometry method. An increasing shear rate can reduce WAT. Figure 4.1, Figure 4.2 and Table 4.5 show critical temperature points where abrupt change of the viscosity slope occurs for three different shear rates. The deposited wax weight at temperatures 55°C and 45°C are 0.6299 g and 1.1641 g, respectively. The wax become completely solid at 35°C by cold finger method. The deposited amount increases with lowering the temperature until its pour point. This test results show that there is no significant variations with tests (Min, 2018).

Table 4. 1 Pour point of Fang crude oil

	Pour point
	(°c)
Fang oil field crude oil	36

Table 4. 2 Wax appearance temperature (WAT) of Fang crude oil

	WAT (°c)		
	6/S	12/S	24/S
Fang oil field crude oil	44.5	44.5	42.5

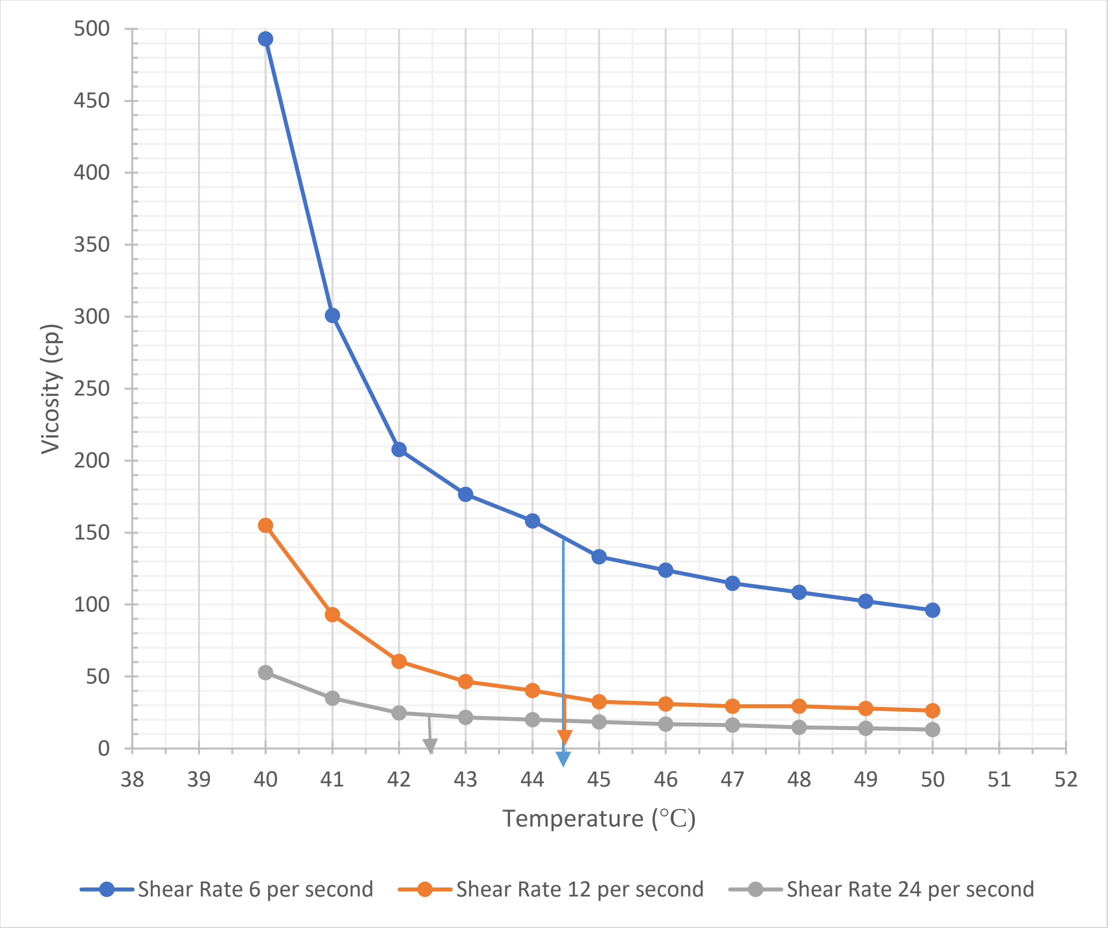


Figure 4. 1 WAT of Fang crude oil with 6/s,12/s and 24/s shear rates.

Table 4. 3 Average deposited wax weight (g) of Fang crude oil

Average deposited wax weight (g)			
	55°C	45°C	35°C
Fang oil field crude oil	0.6299	1.1641	N/A

Regression analysis of WAT test and average deposited wax weight test of crude oil in Fang condition has done. Those regression analysis equations in Table 4.4 can predict WAT at different shear rates. It can also predict wax deposited weight amount at different temperature of crude oil in Fang.

Table 4. 4 Regression analysis of WAT and average deposited wax weight tests of Fang crude oil

Test	Regression Equation
WAT test	*From Shear Rate (γ) 6/s to 24/s*
	$\mathrm{WAT}\ (^\circ\mathrm{C}) = -2.164 \ln(\gamma) + 49.377$
Avg. deposited wax weight test	*From Temperature (T) 35°C to 55°C*
	$\text{Avg. Deposited Wax Weight (g)} = -1.851 \ln(T) + 8.0953$

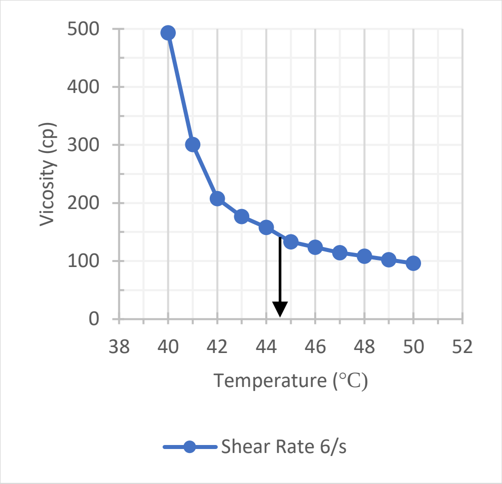


Figure 4.2 (a)

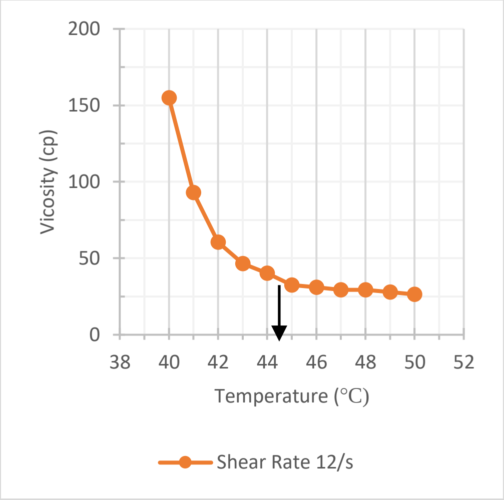


Figure 4.2 (b)

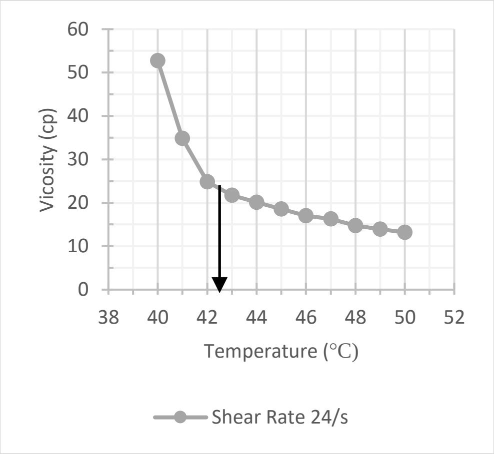


Figure 4.2 (c)

Figure 4. 2 WAT of crude oil at Fang oil field at (a) 6/s, (b)12/s and (c) 24/s shear rates.

Table 4. 5 Slopes of the measured viscosities vs temperature of Fang crude oil in each shear rate

T (°C)	6/s	12/s	24/s
50-49	-6.18	-1.55	-0.8
49-48	-6.2	-1.55	-0.8
48-47	-6.2	0	-1.6
47-46	-9.3	-1.55	-0.8
46-45	-9.3	-1.55	-1.6
45-44	**-24.8**	**-7.75**	-1.6
44-43	-18.6	-6.2	-1.6
43-42	-31	-14	**-3.1**
42-41	-93.1	-32.6	-10
41-40	-192	-62	-18
WAT	44.5	44.5	42.5

4.2 Effect of Hexane on Pour Point, WAT and Wax Deposition Tests

4.2.1 Effect of hexane on pour point tests

The results from pour point tests are illustrated in Figure 4.3 and Table 4.6. It can clearly see that the pour point temperature is reducing with increasing hexane concentration (Numura, 2005) (Farazmand et al., 2016) (Min & Maneeintr, 2019). Significantly reduction of pour point temperature can see at higher hexane concentration which are 15% and 20%. Hexane at 5%,10%,15% and 20% give attribute point 33°C, 31°C, 26°C and 19°C, respectively. Pour point was reduced 47% from original condition with hexane 20%.

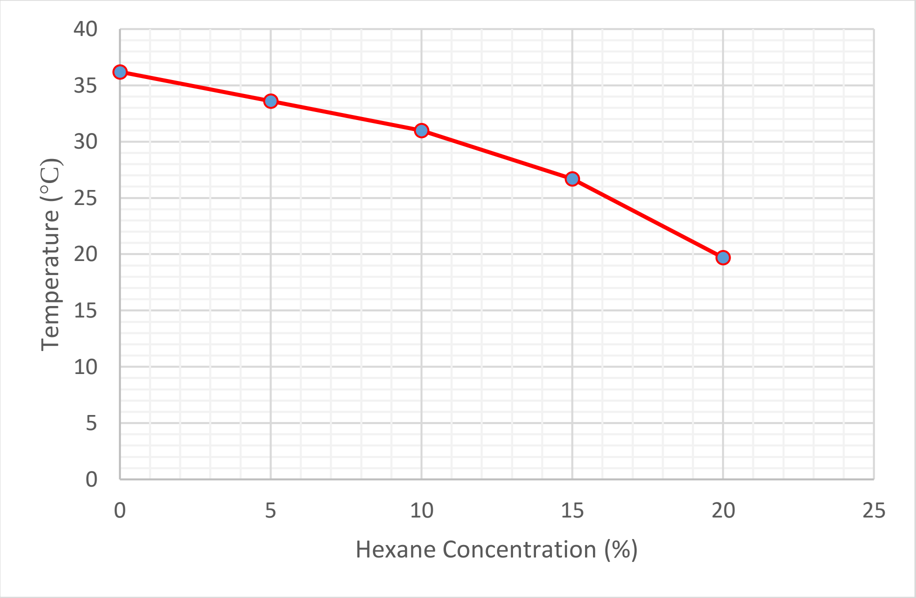


Figure 4. 3 Effect of hexane concentrations on pour point tests

Table 4. 6 Results of increasing hexane concentrations on pour point tests

Hexane	Pour point
%	°c
0	36
5	33
10	31
15	26
20	19

The following non-linear regression equation has developed from pour point test results:

Table 4. 7 Regression analysis of pour points of Fang crude oil with different hexane concentrations

Pour point test conditions	
Hexane 5% to 20%	Pour point (°C) = -9.279 ln (CONC.%) + 50.057

Those regression equations can predict pour point under different conditions for Fang crude oil by using hexane and other interested solvents.

4.2.2 Effect of hexane on wax appearance temperature (WAT) tests

Figure 4.4 to Figure 4.7 show the results of wax appearance temperature (WAT) tests with 5% to 20% hexane at 3 different shear rates 6/s,12/s and 24/s.

As shown in Table 4.8, WAT decreases with increasing share rate from 6/s to 24/s, or hexane concentration increases. But when hexane is fixed in the range from 0% to 5%, WAT does not reduce at shear rates 12/s and 24/s. WAT is decreased from 44.5°C to 43.5°C, from 43.5°C to 42.5°C and from 42.5°C to 41.5°C at shear rate 6/s, 12/s and 24/s with increasing hexane concentration from 10% to 15%. WAT has decreased to 43.5°C in all shear rate 6/s, 12/s and 24/s at 20% hexane concentration. WATs are the points where the change of slope occur as they can be seen from Appendix G for Figure G.1 through Figure G.4 and Table G.1 through Table G.4, slopes of measured viscosities over temperature points at different shear rates and concentrations. From analyzing the results, it can be concluded that shear rate effect cannot see in lowering the WAT at the highest hexane concentration that was tested, 20%. 2.2% of WAT is reduced from original condition with shear rate not only 6/s but also 12/s at hexane 20%. The inhibitor concentration, shear rate, cooling rate and temperature are factors that affect wax crystal growth development (Jennings &

Weispfennig, 2005) (Ridzuan et al., 2015) (Chi et al., 2017) (Ruwoldt et al., 2018) (Min & Maneeintr, 2019).

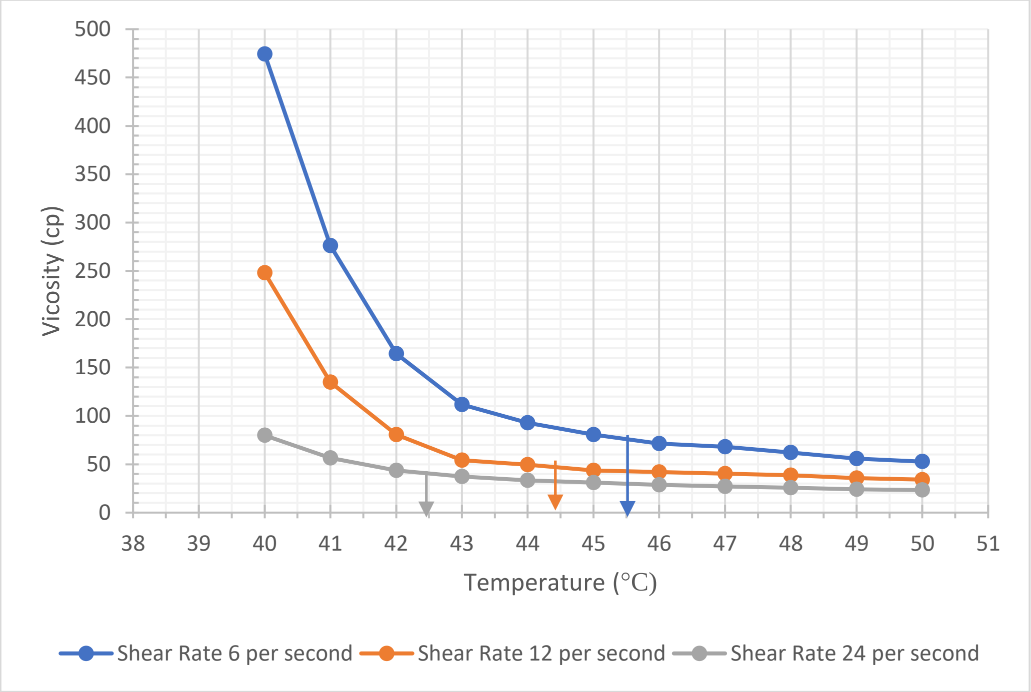


Figure 4. 4 Effect of shear rates on WAT tests at 5% hexane.

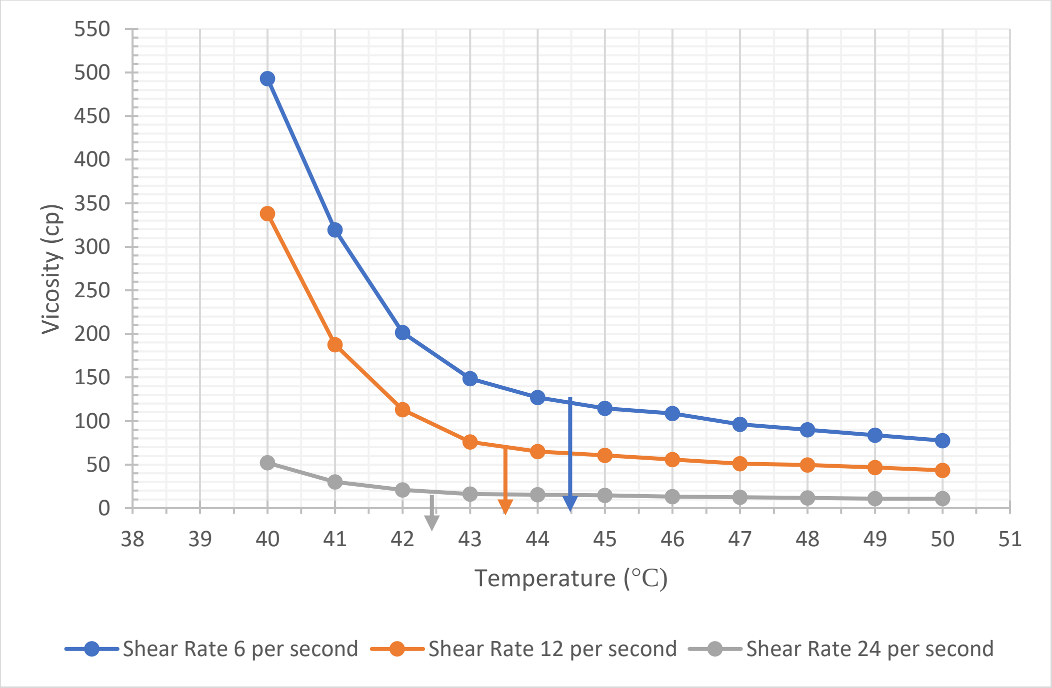


Figure 4. 5 Effect of shear rates on WAT tests at 10% hexane.

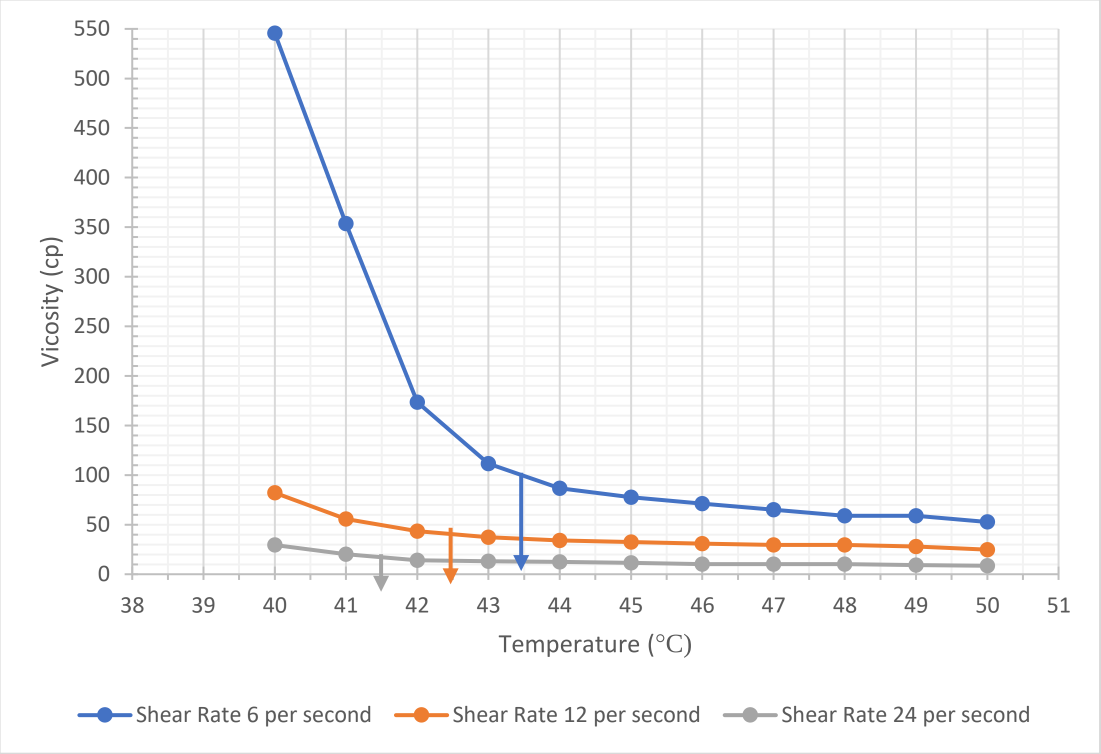


Figure 4. 6 Effect of shear rates on WAT tests at 15% hexane.

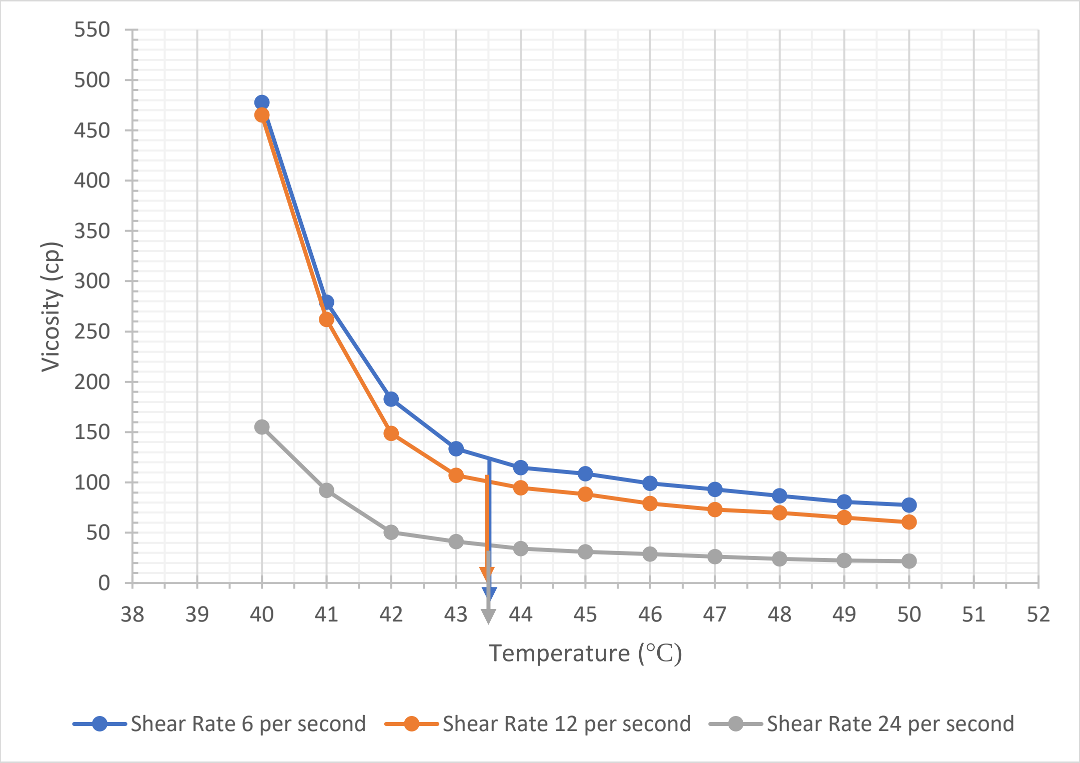


Figure 4. 7 Effect of shear rates on WAT tests at 20 % hexane.

Table 4. 8 Results of increasing hexane concentrations on WAT tests

Hexane	WAT (°c)		
%	6/S	12/S	24/S
0	44.5	44.5	42.5
5	45.5	44.5	42.5
10	44.5	43.5	42.5
15	43.5	42.5	41.5
20	43.5	43.5	43.5

4.2.3 Effect of hexane on wax deposition tests

The results of hexane concentrations on deposited wax weight test as a function of concentrations and temperature are can be seen in Figure 4.8 and Figure 4.9. Test period can also influence deposited weight amount (Kasumu & Mehrotra, 2015). Hexane can dilute the paraffin wax components since it is one of the light hydrocarbon solvents. During 3-hours tests, it is obvious that hexane at high concentration can reduce deposited wax weight significantly (Ridzuan et al., 2016), as seen in both Table 4.9 and Figure 4.8. With hexane 20%, average deposited wax weight is reduced to 0.2045 g at 55°C, 0.2168 g at 45°C and 0.4545 g at 35°C, respectively. With hexane 20%, 81.38% of wax weight is reduced from original condition at 45°C temperature while 67.54% is reduced at 55°C. Temperature during tests is another influencing factor on deposited weight tests (Jennings & Weispfennig, 2005). Higher temperature can reduce deposited wax weight obviously (Ruwoldt et al., 2018) (Min, 2018) in each concentration, as shown in Table 4.9. At 35°C, hexane 10%,15% and 20% give 1.2874 g, 0.6608 g and 0.4545 g respectively. But hexane 5%,10%,15% and 20% give decrease deposited amount 0.5570 g, 0.3184 g, 0.2795 g and 0.2045 g at 55°C. When increase temperature from 35°C to 45°C with hexane 20%, 52.3% of wax weight is reduced. 55.01% was lowered when increase from 35°C to 45°C.

Table 4. 9 Results of increasing hexane concentrations on wax deposition tests

Average deposited wax weight (g)			
Hexane %	at 55°C	at 45°C	at 35°C
0	0.6299	1.1641	N/A
5	0.5570	0.7496	N/A
10	0.3184	0.3381	1.2874
15	0.2795	0.2938	0.6608
20	0.2045	0.2168	0.4545

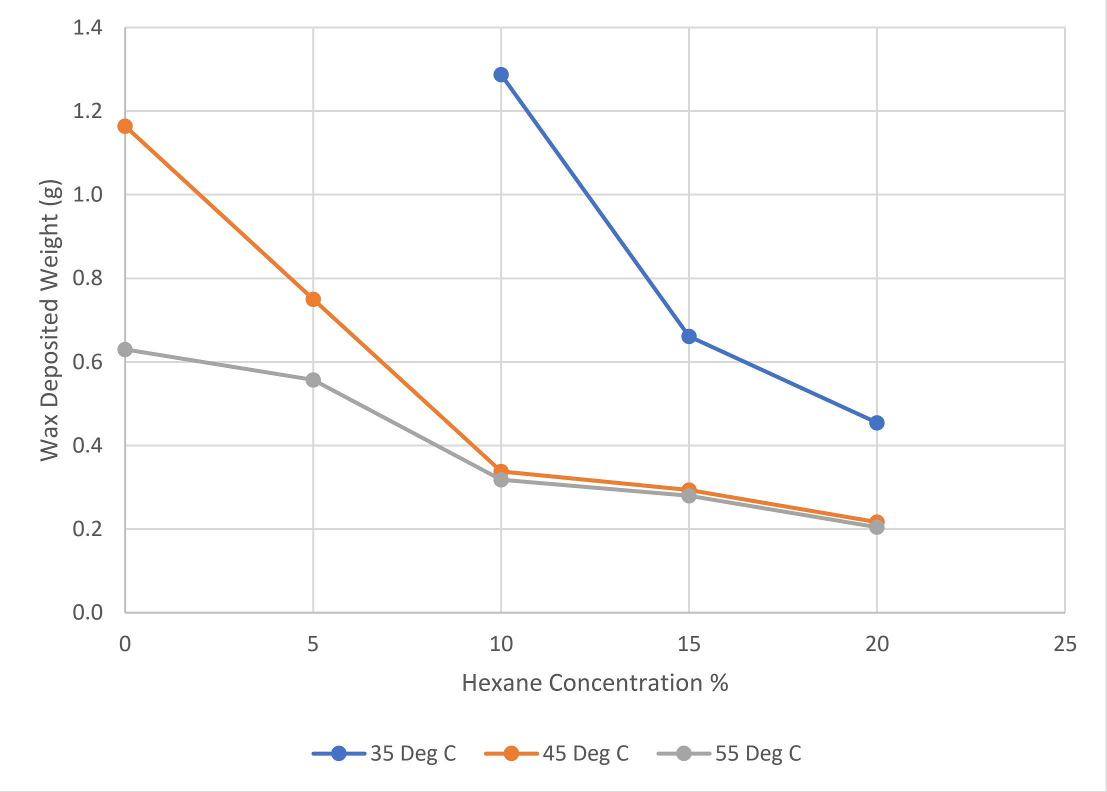


Figure 4. 8 Effect of hexane concentrations on wax deposition tests

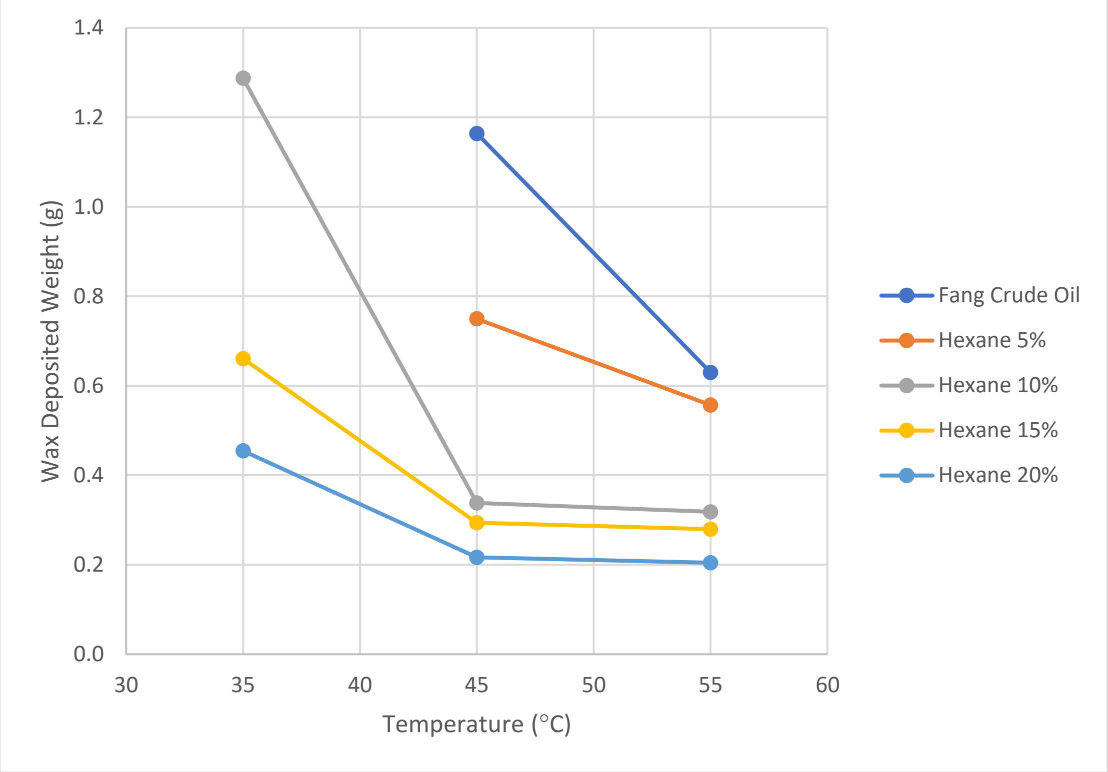


Figure 4. 9 Effect of temperature on wax deposition tests

4.3 Calculating Wax Appearance Temperature (WAT)

The intersection of viscosities calculated by two equations, Eq. 3.1 and Eq.3.2, will gives calculated wax appearance temperature (WAT) in Fang crude oil condition at shear rate 6/s,12/s and 24/s. The results are shown from Figure 4.10 to Figure 4.12. Non-linear regressions of the lab data have coefficients of determination (R^2) greater than 0.9, as shown in Appendix E in higher and lower temperature regions of the viscosity and temperature curves. The slopes of the lab data remain constant for higher temperature region, but lab data slope increase in lower temperature region because the precipitation of wax which increases the dispersed phase volume fraction (Farah et al., 2005). The results of calculated and experimental WAT with their percentage of difference are shown in Table 4.10. By increasing shear rate and hexane concentration make calculated WAT lower which can also be seen in Figure 4.13. The figures of calculated WATs in hexane concentrations 5%,10%,15% and 20% at shear rates 6/s,12/s and 24/s can be seen in Appendix F.

The calculated WAT of 44°C attained by intersection of higher temperature equation Eq. 3.1 and lower temperature equation Eq. 3.2 in Fang crude oil at shear rate 6/s as seen in Figure 4.10 and Table 4.10. Coefficients, c_1, c_2, c_3 and c_4, and Eq. 3.1 and Eq. 3.2 under different hexane concentrations, 0%, 5%, 10%, 15% and 20%, with shear rates 6/s, 12/s and 24/s, can be seen in Appendix E. Also, viscosities achieved from experimental results are compared with viscosities attained from two equations in through the Figures. Also the calculated WAT, 44°C and 42.6°C, of Fang crude oil with shear rate 12/s and 24/s are shown in Figure 4.11 and Figure 4.12.

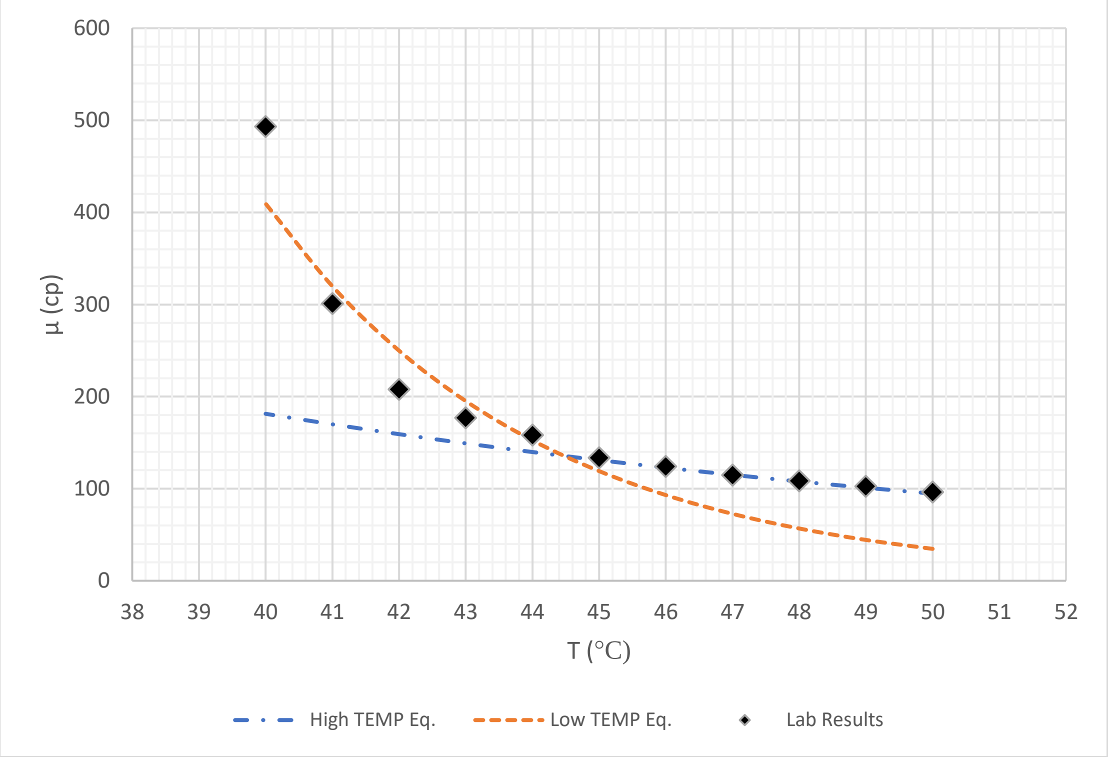


Figure 4. 10 Calculated viscosities result of Fang crude oil at shear rate 6/s

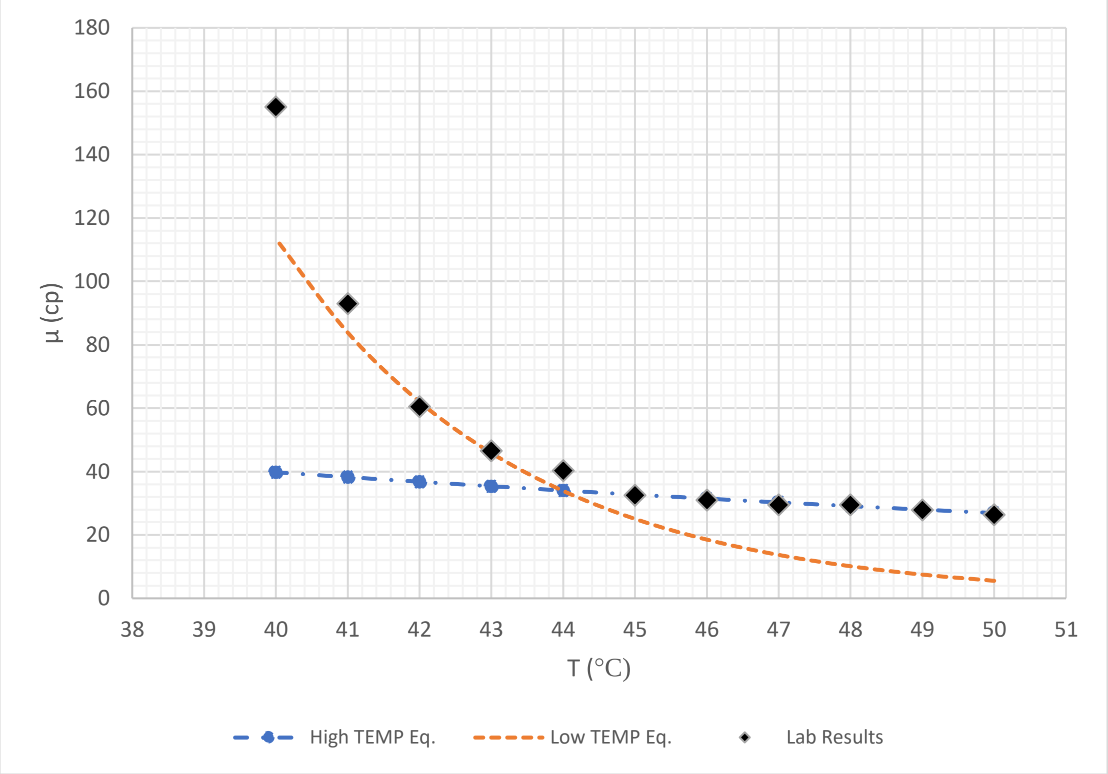


Figure 4. 11 Calculated viscosities result of Fang crude oil at shear rate 12/s

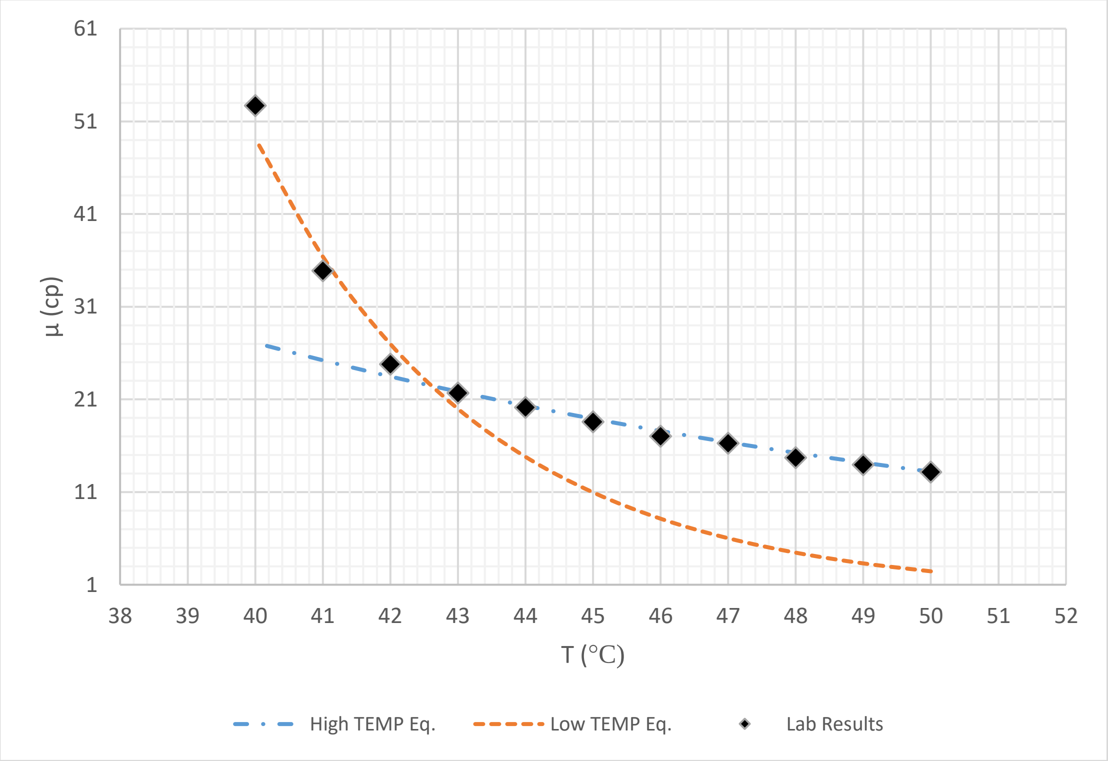


Figure 4. 12 Calculated viscosities result of Fang crude oil at shear rate 24/s

Table 4. 10 Calculated WAT results of Fang crude oil and crude oil with difference hexane concentrations

Condition	γ 6/S			γ 12/S			γ 24/S		
	EXP.	CALC.	% diff	EXP.	CALC.	% diff	EXP.	CALC.	% diff
Oil	44.5	44.4	0.2	44.5	44	1.1	42.5	42.6	-0.2
Hexane 5%	45.5	44.4	2.4	44.5	44	1.1	42.5	42.6	-0.2
Hexane 10%	44.5	43.6	2.0	43.5	43.6	-0.2	42.5	42.2	0.7
Hexane 15%	43.5	43.4	0.2	42.5	43.4	-2.1	41.5	42	-1.2
Hexane 20%	43.5	43.4	0.2	43.5	43.2	0.7	43.5	42	3.4

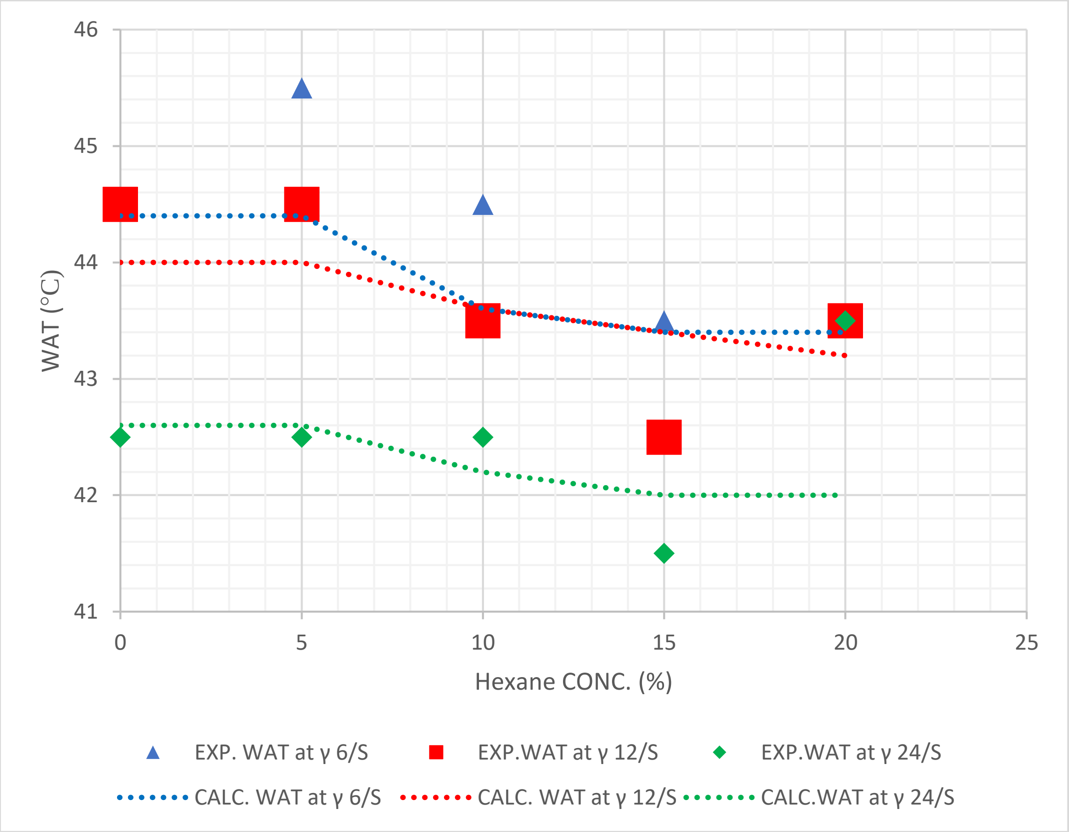
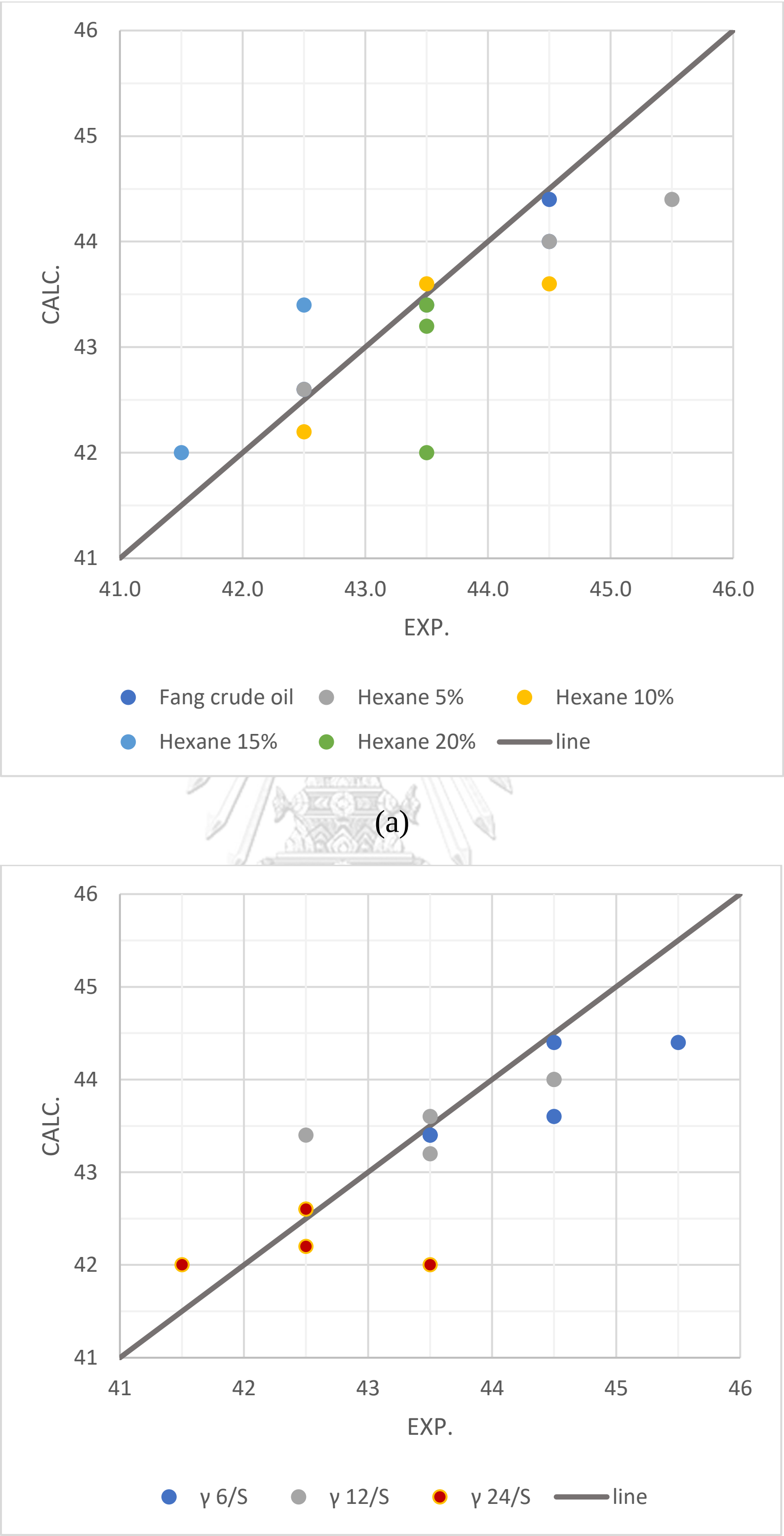


Figure 4. 13 Calculated wax appearance temperatures (WAT) of Fang crude oil and crude oil with difference hexane concentrations

As shown in Table 4.10, the calculated WAT at shear rate 24/s has reduced to 42.6°C for hexane at 0% and at 5%, 42.2°C in hexane 10%, 42°C in hexane 15% and 20%. The calculated WAT at shear rate 12/s has reduced to 44°C in hexane 0% and 5%, 43.6°C in hexane 10%, 43.4°C in hexane 15% and 43.2°C in hexane 20%. The calculated WAT at shear rate 6/s has reduced to 44.4°C in hexane 0% and 5%, 43.6°C in hexane 10%, 43.4°c in hexane 15% and 20%. The experimental WAT and calculated WAT parity plot can be seen in Figure 4.14. Selected equations can apply in perceptive of both different concentrations and shear rates. The percentage average absolute deviation, AAD, for those two is 1.08 %.



(a)

(b)

Figure 4. 14 Experimental versus calculated wax appearance temperature (WAT)

4.4 Results from Wax Deposition Correlations

The results of calculated wax weight by modified Ronningsen's correlation are shown in Table 4.11. Effect of hexane concentration and temperature on calculated wax weight by this correlation is also shown in Figure 4.15 and Figure 4.16.

When hexane concentration increases from 0% to 20% in temperatures (55°C, 45°C and 35°C), the calculated deposited wax weight is obviously reduced. In each of the concentration, the temperature of the highest i.e. 55°C gives the lowest calculated result, 0.6283 g, 0.4545 g, 0.3287 g, 0.2378 g and 0.1720 g in Hexane 0%, 5%, 15% and 20%, receptively. The percent difference (% diff) is also found between experience and calculated wax weight results. The parity plot of this correlation is shown in Figure 4.17 and average absolute deviation for percentage difference is 17.01%. This correlation result is fit well with experimental results as shown in Figure 4.17. The hexane concentration effect on calculated wax weight results are compare and shown in Figure 4.15. Figure 4.16 shows temperature effect on calculated wax weight with lab results in each hexane concentration.

Table 4. 11 Results of calculated wax weight by modified Ronningsen's correlation

		Modified Ronningsen's correlation		
TEMP.	Hexane CONC.	EXP. Wax Weight	CALC. Wax Weight	% diff
°C	%	g	g	
55	0	0.6299	0.6283	0.2497
	5	0.5570	0.4545	18.4047
	10	0.3184	0.3287	-3.2480
	15	0.2795	0.2378	14.9240
	20	0.2045	0.1720	15.8934
45	0	1.1641	1.1600	0.3487
	5	0.7496	0.8013	-6.9018
	10	0.3381	0.5535	-63.7234
	15	0.2938	0.3824	-30.1504
	20	0.2168	0.2641	-21.8370
35	10	1.2874	0.9321	27.5991
	15	0.6608	0.6149	6.9460
	20	0.4545	0.4057	10.7477

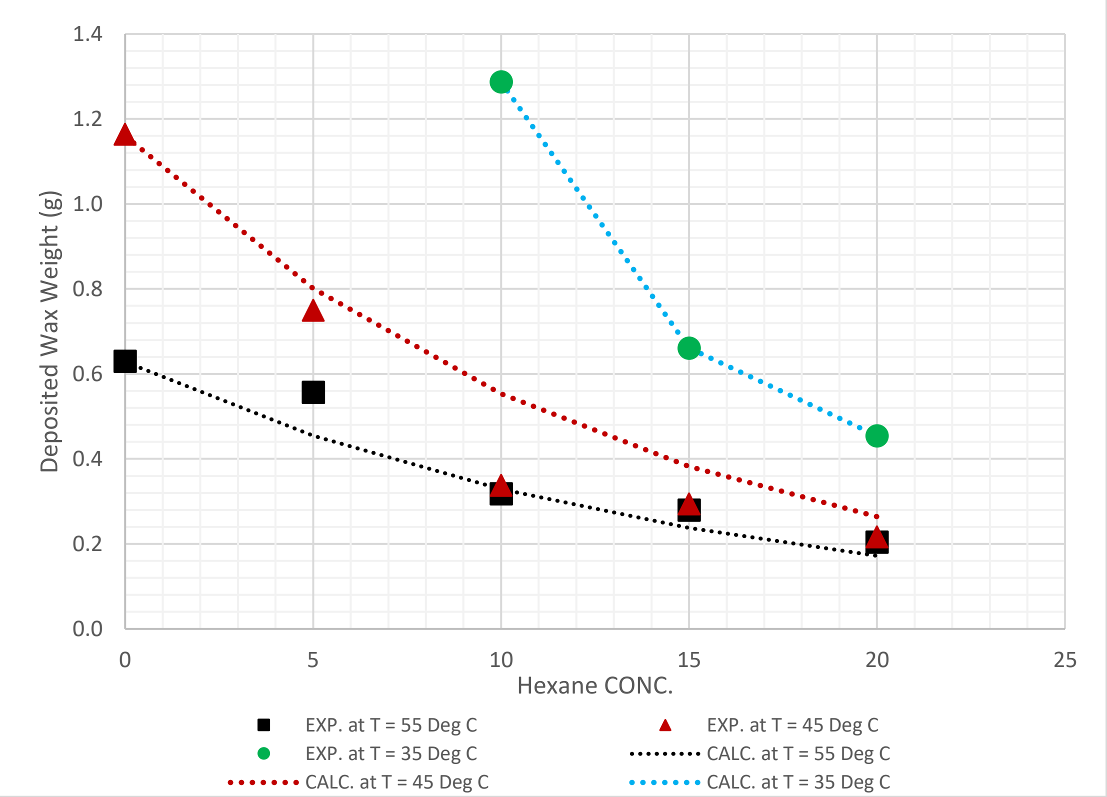


Figure 4. 15 Effect of hexane concentration on calculated wax weight by modified Ronningsen’s correlation

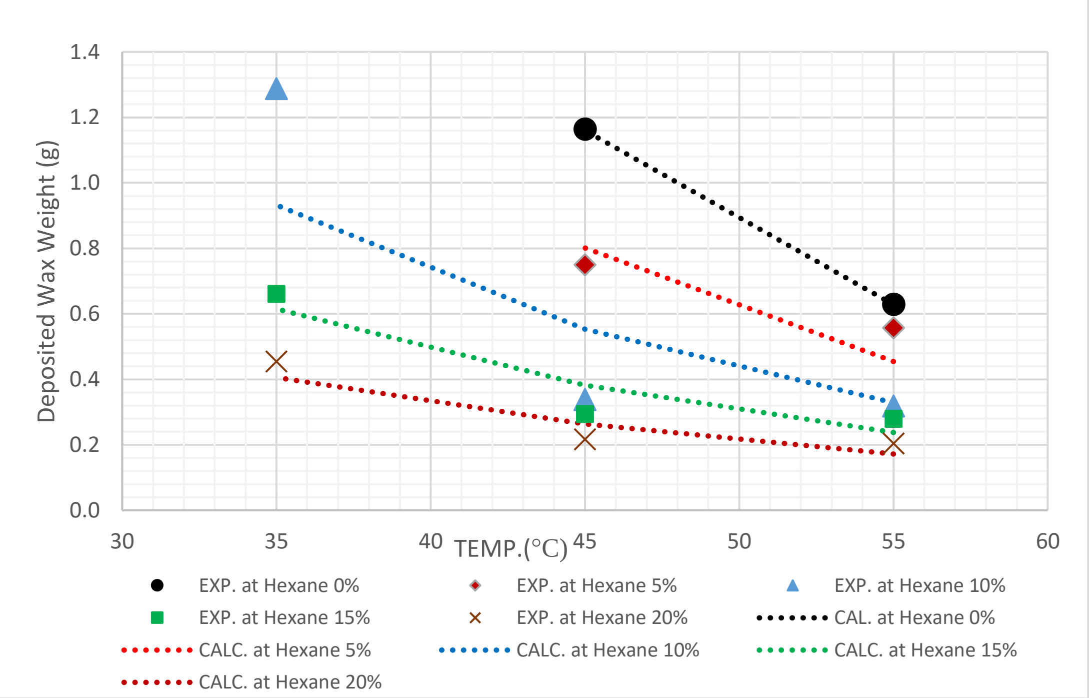
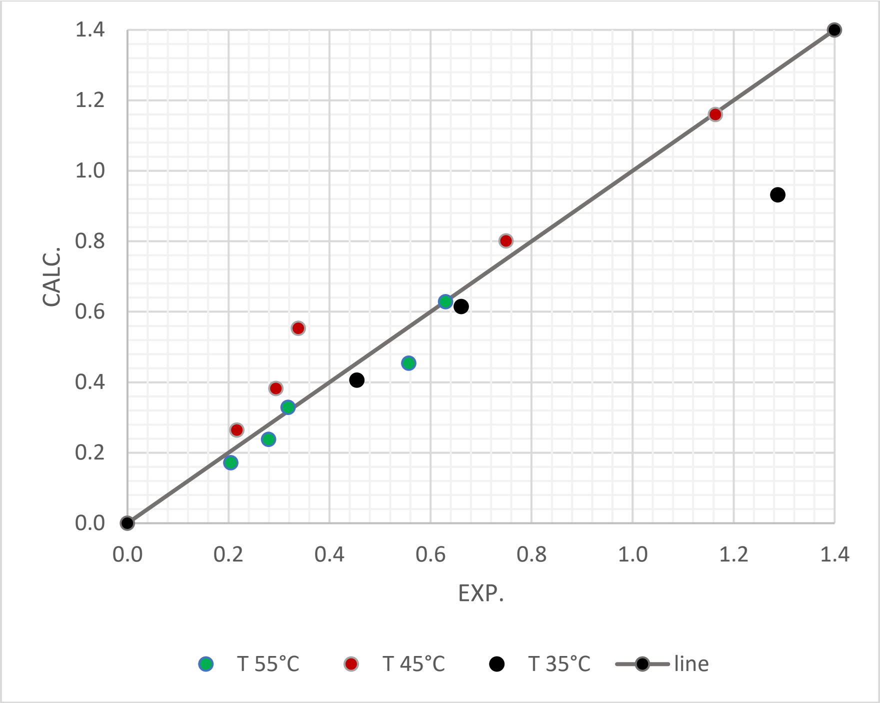
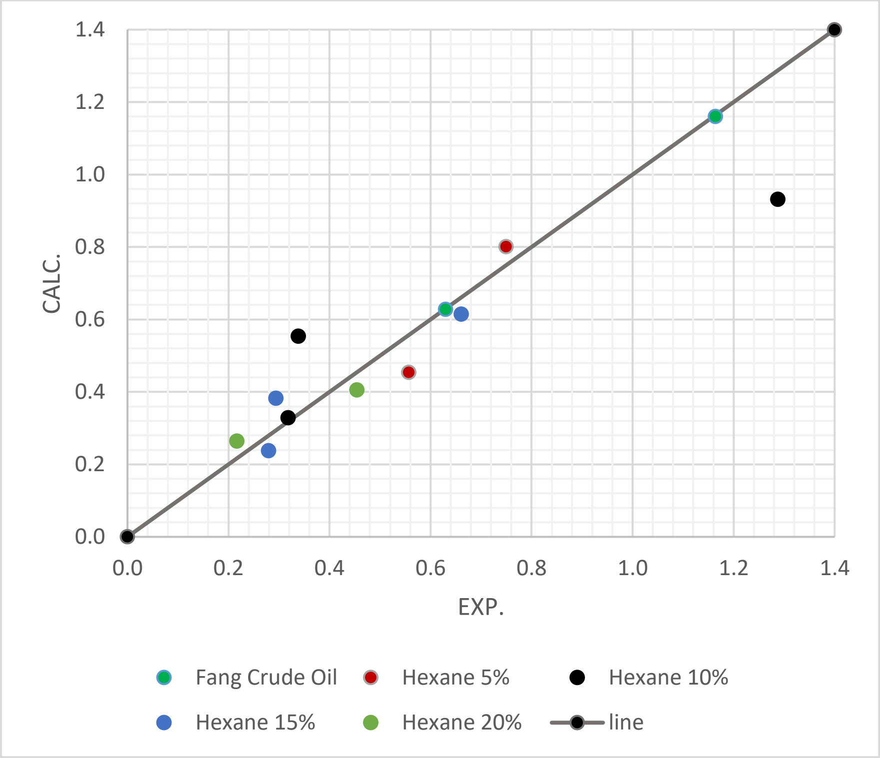


Figure 4. 16 Effect of temperature on calculated wax weight by modified Ronningsen’s correlation



(a)



(b)

Figure 4. 17 Average deposited wax weight of experimental vs modified Ronningsen's correlation

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 Conclusions

The pour point test, the WAT test and wax deposition test are performed for crude oil at Fang conditions at different hexane concentrations. The testing parameters are shear rate, temperature and concentration of hexane with constant cooling rate and time. The correlations are developed from literature review and used in Fang condition. The predicted viscosities are calculated as a function of temperature, shear rates and chemical concentrations to find calculated WATs. The wax deposition correlation is modified from Ronningsen's correlation. The percent difference has made for both lab and calculated WAT and wax deposition amount. The regression analyses are applied on the different test conditions.

Conclusion from this research are present as follows;

1. Among three test results, the WAT test results at lower shear rates are slightly lower than previous work, 44.5°C in both shear rate 6/s and 12/s and 42.5°C in shear rate 24/s. The non-linear regression analysis for both WAT and wax deposition test of Fang crude oil has been made. The regression equation for WAT and wax deposition can predict those parameters under different conditions for Fang crude oil.
2. The higher hexane concentration can reduce pour point. The hexane concentration at 20% gives a significantly pour point reduction at 19°C, 16°C reduction from Fang crude oil original pour point 36°C. The hexane concentration at 15%, 10% and 5% gives 10°C, 5°C and 3°C reduction from original condition, respectively. With hexane 20%, 47% of pour point was reduced from original crude oil pour point. The non-linear regression analysis has done for pour point tests with different hexane concentrations. The regression equations can be applicable with hexane and other interested solvents under different condition for pour point prediction.
3. A constant cooling rate 12°C/hour has been imposed to all WAT tests. There are three different shear rates; 6/s, 12/s and 24/s, with four different Hexane concentrations 5%, 10%, 15% and 20%. The more Hexane concentration was used, the higher WAT reduction was observed. Using Hexane concentration 20% and 15% show the same WATs at 6/s while Hexane 10% and 5% give not significant variation of WATs. At hexane 20%, WAT is reduced to 2.2% with shear rate 6/s and 12/s from original crude oil condition. It is observed that shear rate and concentration percentage play important role in WAT test.

4. The wax deposition tests are processed in the constant duration time. It’s observed that both higher hexane concentration and temperature give the less amount of wax deposit. The deposited wax weight cannot be observed on the temperature lower than 35°C because of the solid condition. Both hexane concentration and temperature are the factors that influences deposited wax. At 55°C, 67.53% of wax was reduced with 20% hexane from original condition meanwhile 81.38% at 45°C. We can see that effect of hexane concentration more at low temperature when reducing wax weight.
5. The coefficients are found from the higher temperature and lower temperature region of lab results. The temperature, shear rates and chemical concentrations are influencing factors for that coefficients. The viscosity can be found by using the correlation equations and those coefficients. The WAT can be obtained at the intersection of two equations. Both increasing Hexane concentration percent and shear rate make calculated WAT decrease. The percent difference is also done for those calculated WAT with experimental WAT. The selected equations can be applied in both concentration and shear rate perceptive by seeing from the parity plot.
6. Modified Roningsen’s work was applied to predict wax deposition amount in different hexane concentrations and temperature. Coefficients for equations are acquired from multiple regression analysis of wax deposition test results. It can be concluded that increasing hexane concentration and decreasing temperature can lower wax deposition amount. Predicted Wax Amount is compared with Experimental Results. Percentage difference between predicted and experimental results has been presented in this research. Results show that this correlation works well with both different solvent concentrations and temperatures.

5.2 Recommendation

More chemical testing for solvents are encouraged to lower the pour point, WAT and deposited amount from crude oil at Fang condition at low concentration, cooling rate and time effect on previous three parameters and emulsion effects on Fang crude oil wax deposition are recommended for further study.

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APPENDIX

A. Fang crude oil sample data

The viscosity of oil is 34 cp at 70°C and the density of oil is 0.85 g/cm3 with the acid number of 0.89 mg KOH/g. The composition of oil ranges from C7 to C35+ alkane and the distribution are as shown in following Table (Saengnil, 2015):

Table A.1 Oil sample composition

(Saengnil, 2015)

Component	weight (%)
C7	0.05
C8	0.68
C9	0.93
C10	1
C11	1.45
C12	1.84
C13	3.06
C14	3.52
C15	4.86
C16	3.87
C17	4.71
Pristane ($C_{19}H_{40}$)	2.44
C18	3.49
Phytane ($C_{20}H_{42}$)	0.82
C19	3.89
C20	4.41
C21	4.81
C22	4.48

C23	4.97
C24	4.26
C25	4.42
C26	4.33
C27	4.56
C28	3.58
C29	3.97
C30	3.72
C31	3.27
C32	2.87
C33	3.64
C34	1.7
C35+	4.4

Table A.2 Density of crude oil sample

(Saengnil, 2015)

Temperature (°C)	Oil Density (g/cm3)
70	0.85023
80	0.84885
90	0.84747

B. Pour point test

Test Type: Pour Point Test

Method: ASTM D 5853-11

Instrument: Julabo Immersion circulator model heating or cooling bath machine

Kimo thermocouple data logger’s model

Thermocouple J-Type

Water as heating and cooling media

Test Tube

Condition: Crude oil at Maesoon condition

Crude oil with 5%,10%,15% and 20 % concentrations of Hexane

Temperature: from 65°C to until pour point Temperature of each condition

Time: Depends on Condition

Results: Pour Point of Crude oil at Maesoon condition

Pour Point Crude oil with 5%,10%,15% and 20% concentrations of Hexane

C. Wax appearance temperature (WAT) test

Test Type:	Wax Appearance Temperature (WAT) test
Method:	Viscometery
Instrument:	DV2TLV Viscometer Julabo F26 model heating or cooling bath machine Glycol as heating and cooling media Spindle Number 52
Condition:	Crude oil at Maesoon condition Crude oil with 5%,10%,15% and 20 % concentrations of Hexane
Shear Rates:	6/s,12/s & 24/s
Temperature:	from 80 °C to 40 °C
Time:	Approximate 7 to 8 Hours
Results:	WAT of Crude oil at Maesoon condition WATs of Crude oil with 5%,10%,15% and 20% concentrations of Hexane

D. Wax deposition test

Test Type:	Wax Deposition Test
Method:	Cold Finger
Instrument:	Julabo Immersion circulator model heating or cooling bath machine
	Kimo thermocouple data logger’s model
	Thermocouple J-Type
	Water as heating and cooling media
	the precision 4 digits weighing machine
	Test Tubes
Condition:	Crude oil at Maesoon condition
	Crude oil with 5%,10%,15% and 20 % concentrations of Hexane
Temperature:	at 55°C, 45°C & 35°C
Time:	3 hours for each temperature condition
Results:	Average Deposited Wax Weight Amount of Crude oil at
	Maesoon condition
	Average Deposited Wax Weight Amount of Crude oil with 5%,10%,15% and 20% concentrations of Hexane

E. Coefficients for high TEMP Eq. and low TEMP Eq.*

Condition	Equation	T Range (°C)		Coefficient		R^2
Fang Crude Oil						
Shear Rate 6/s	+High TEMP.	From	50	c_1	2441	0.9964
		To	45	c_2	-0.065	
	++Low TEMP.	From	45	c_3	8.00E+06	0.917
		To	40	c_4	-0.247	
Shear Rate 12/s	+High TEMP.	From	50	c_1	189.22	0.9644
		To	45	c_2	-0.039	
	++Low TEMP.	From	45	c_3	2.00E+07	0.9466
		To	40	c_4	-0.302	
Shear Rate 24/s	+High TEMP.	From	50	c_1	482.82	0.9954
		To	43	c_2	-0.072	
	++Low TEMP.	From	43	c_3	8000000	0.9568
		To	40	c_4	-0.3	
Hexane 5%						
Shear Rate 6/s	+High TEMP.	From	50	c_1	3555.6	0.9886
		To	45	c_2	-0.084	
	++Low TEMP.	From	45	c_3	7.00E+08	0.9383
		To	40	c_4	-0.358	
Shear Rate 12/s	+High TEMP.	From	50	c_1	404.37	0.9775
		To	45	c_2	-0.049	
	++Low TEMP.	From	45	c_3	2.00E+08	0.9108
		To	40	c_4	-0.346	
Shear Rate 24/s	+High TEMP.	From	50	c_1	617.68	0.9821
		To	43	c_2	-0.066	
	++Low TEMP.	From	43	c_3	2.00E+06	0.9729
		To	40	c_4	-0.256	
Hexane 10%						
Shear Rate 6/s	+High TEMP.	From	50	c_1	4675.9	0.9941
		To	44	c_2	-0.082	
	++Low TEMP.	From	44	c_3	5.00E+08	0.9683
		To	40	c_4	-0.347	
Shear Rate 12/s	+High TEMP.	From	50	c_1	1192.7	0.9899
		To	44	c_2	-0.066	
	++Low TEMP.	From	44	c_3	5.00E+09	0.9648
		To	40	c_4	-0.416	
Shear Rate 24/s	+High TEMP.	From	50	c_1	258.05	0.9766
		To	43	c_2	-0.064	

	++Low TEMP.	From	43	c_3	200000000	0.9722
		To	40	c_4	-0.385	
Hexane 15%						
Shear Rate 6/s	+High TEMP.	From	50	c_1	6295.3	0.9339
		To	43	c_2	-0.096	
	++Low TEMP.	From	43	c_3	2E+12	0.9901
		To	40	c_4	-0.547	
Shear Rate 12/s	+High TEMP.	From	50	c_1	270.38	0.9377
		To	44	c_2	-0.047	
	++Low TEMP.	From	44	c_3	420880	0.9152
		To	40	c_4	-0.216	
Shear Rate 24/s	+High TEMP.	From	50	c_1	168.27	0.9598
		To	42	c_2	-0.06	
	++Low TEMP.	From	42	c_3	9.00E+07	0.9999
		To	40	c_4	-0.374	
Hexane 20%						
Shear Rate 6/s	+High TEMP.	From	50	c_1	2279.5	0.9944
		To	44	c_2	-0.068	
	++Low TEMP.	From	44	c_3	7.00E+08	0.9587
		To	40	c_4	-0.359	
Shear Rate 12/s	+High TEMP.	From	50	c_1	3901.7	0.9686
		To	43	c_2	-0.084	
	++Low TEMP.	From	43	c_3	7.00E+10	0.9909
		To	40	c_4	-0.471	
Shear Rate 24/s	+High TEMP.	From	50	c_1	1689.1	0.9675
		To	43	c_2	-0.088	
	++Low TEMP.	From	43	c_3	1E+10	0.9664
		To	40	c_4	-0.459	

$$+\ \mu_{\text{High TEMP. Zone}} = c_1 * e^{c_2 * T} \quad \text{Eq. (3.1)}$$

$$++\ \mu_{\text{Low TEMP. Zone}} = c_3 * e^{c_4 * T} \quad \text{Eq. (3.2)}$$

*Eq. 3.1 with coefficients c_1 and c_2 are used as a Higher Temperature zone Eq. and Eq. 3.2 with coefficient c_3 and c_4 used as a Low Temperature zone Eq. under Hexane concentrations in 0%,5%,10%,15% and 20% at shear rate 6/s,12/s and 24/s to find calculated WAT in Section 4.4 in Chapter 4.

F. Effect of hexane on Fang crude oil WAT

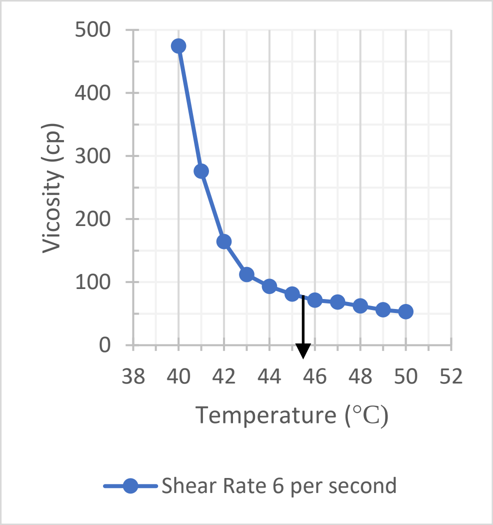

Figure G.1(a)

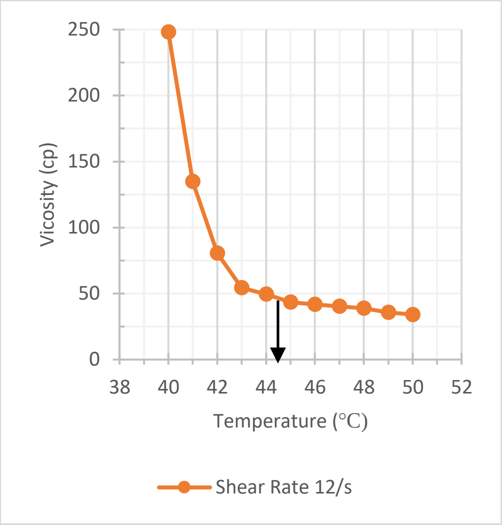

Figure G.1(b)

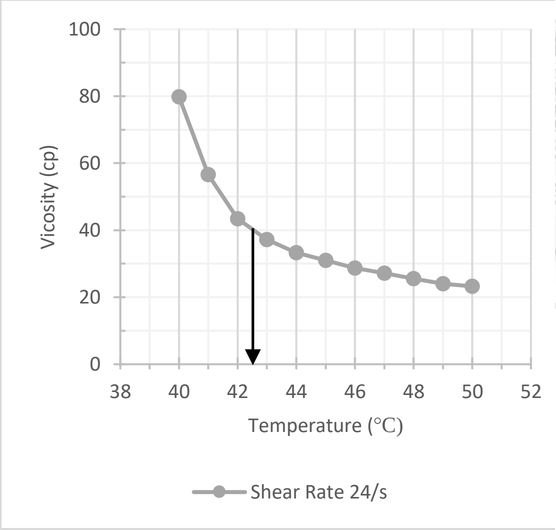

Figure G.3(C)

Figure G.1 WAT of Fang crude oil with hexane 5% at (a) 6/s, (b)12/s and (c) 24/s shear rates.

Table G.1 Slopes of the measured viscosities vs temperature of Fang crude oil with hexane 5% in each shear rate

T (°C)	6/s	12/s	24/s
50-49	-3.1	-1.55	-0.78
49-48	-6.2	-3.1	-1.55
48-47	-6.2	-1.55	-1.55
47-46	-3.1	-1.55	-1.55
46-45	**-9.31**	-1.55	-2.33
45-44	-12.4	**-6.2**	-2.32
44-43	-18.6	-4.65	-3.88
43-42	-52.7	-26.36	**-6.2**
42-41	-112	-54.28	-13.18
41-40	-198	-113.2	-23.25
WAT	45.5	44.5	42.5

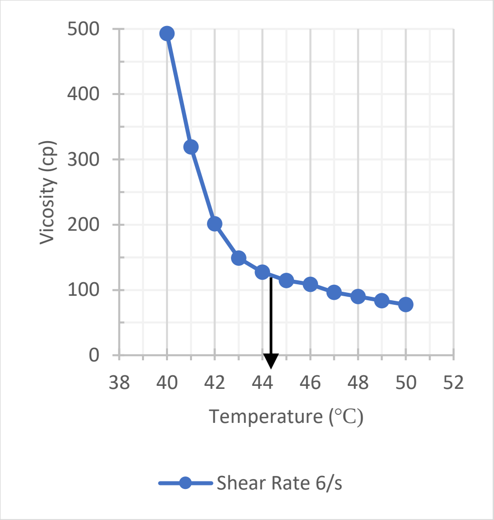

Figure G.2 (a)

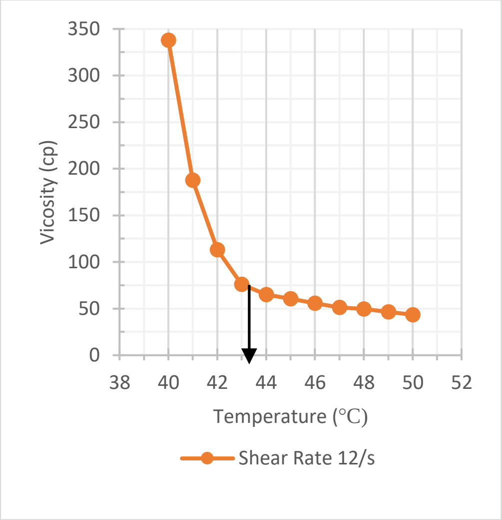

Figure G.2 (b)

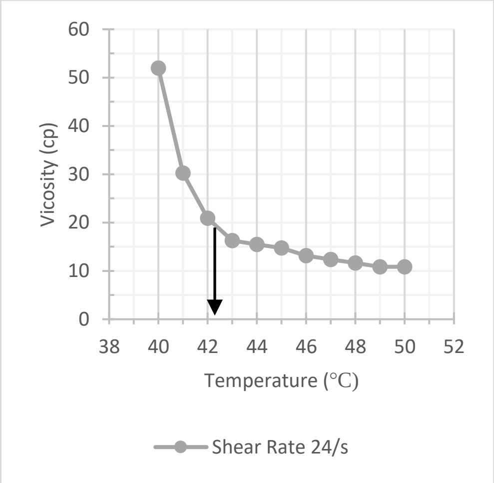

Figure G.2 (C)

Figure G.2 WAT of Fang crude oil with hexane 10% at (a) 6/s, (b)12/s and (c) 24/s shear rates.

Table G.2 Slopes of the measured viscosities vs temperature of Fang crude oil with hexane 10% in each shear rate

T (°C)	6/s	12/s	24/s
50-49	-6.2	-3.1	0
49-48	-6.2	-3.1	-0.78
48-47	-6.2	-1.55	-0.77
47-46	-12.38	-4.65	-0.78
46-45	-6.2	-4.65	-1.55
45-44	**-12.4**	-4.65	-0.77
44-43	-21.7	**-10.86**	-0.78
43-42	-52.7	-37.23	**-4.65**
42-41	-117.9	-74.4	-9.3
41-40	-173.6	-150.4	-21.71
WAT	44.5	43.5	42.5

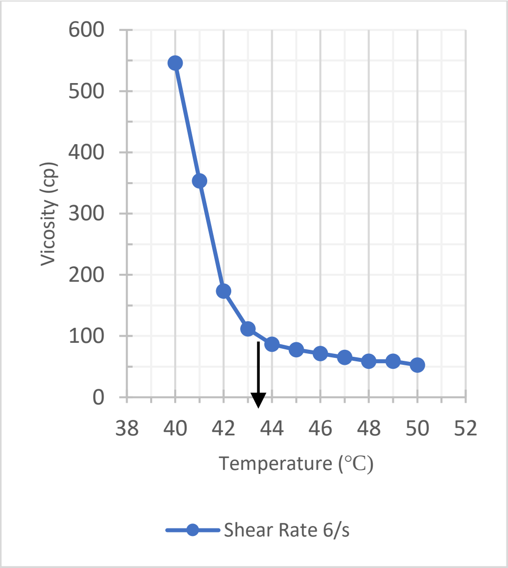

Figure G.3: (a)

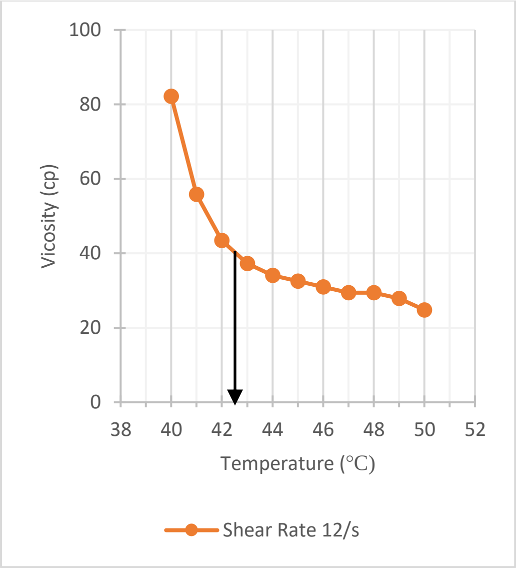

Figure G.3(b)

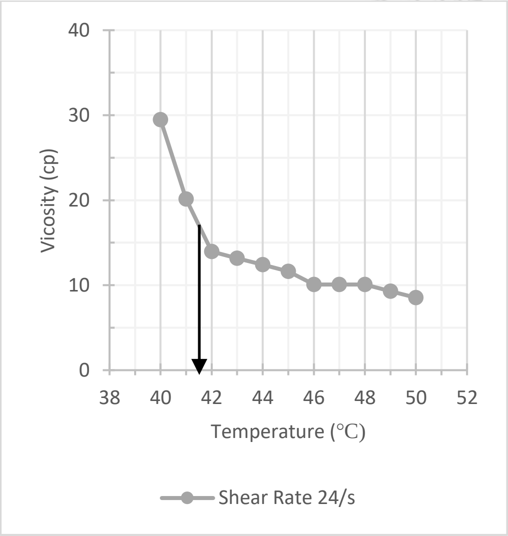

Figure G.4 (C)

Figure G.3 WAT of Fang crude oil with hexane 15% at (a) 6/s, (b)12/s and (c) 24/s shear rates.

Table G.3 Slopes of the measured viscosities vs temperature of Fang crude oil with hexane 15% in each shear rate

T (°C)	6/s	12/s	24/s
50-49	-6.2	-3.1	-0.77
49-48	0	-1.55	-0.78
48-47	-6.2	0	0
47-46	-6.2	-1.55	0
46-45	-6.21	-1.55	-1.55
45-44	-9.3	-1.55	-0.77
44-43	**-24.78**	-3.1	-0.78
43-42	-62	**-6.2**	-0.77
42-41	-179.9	-12.4	**-6.2**
41-40	-192.2	-26.36	-9.31
WAT	43.5	42.5	41.5

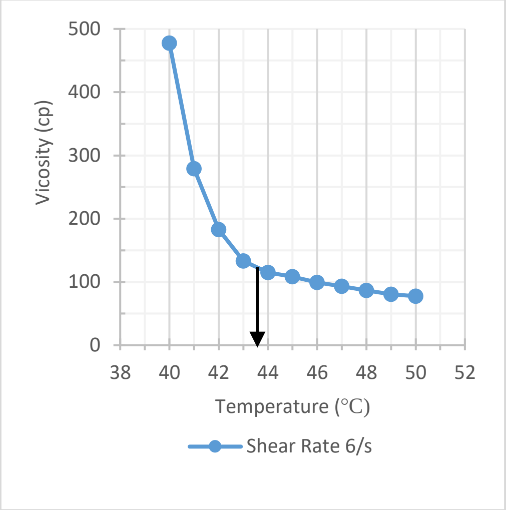

Figure G.4 (a)

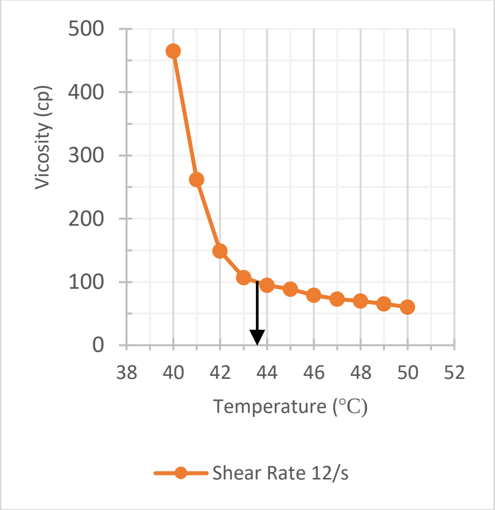

Figure G.4 (b)

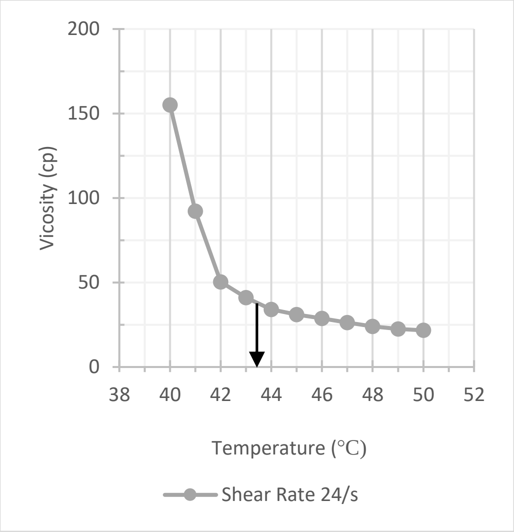

Figure G.4 (c)

Figure G.4 WAT of Fang crude oil with hexane 20% at (a) 6/s, (b)12/s and (c) 24/s shear rates.

Table G.4 Slopes of the measured viscosities vs temperature of fang crude oil with hexane 20% in each shear rate

T (°C)	6/s	12/s	24/s
50-49	-3.1	-4.65	-0.78
49-48	-6.2	-4.65	-1.55
48-47	-6.2	-3.1	-2.33
47-46	-6.2	-6.21	-2.32
46-45	-9.28	-9.3	-2.33
45-44	-6.2	-6.2	-3.1
44-43	**-18.6**	**-12.43**	**-6.97**
43-42	-49.6	-41.8	-9.31
42-41	-96.2	-113.2	-41.85
41-40	-198.4	-203.1	-62.76
WAT	43.5	43.5	43.5

G. Calculated WAT

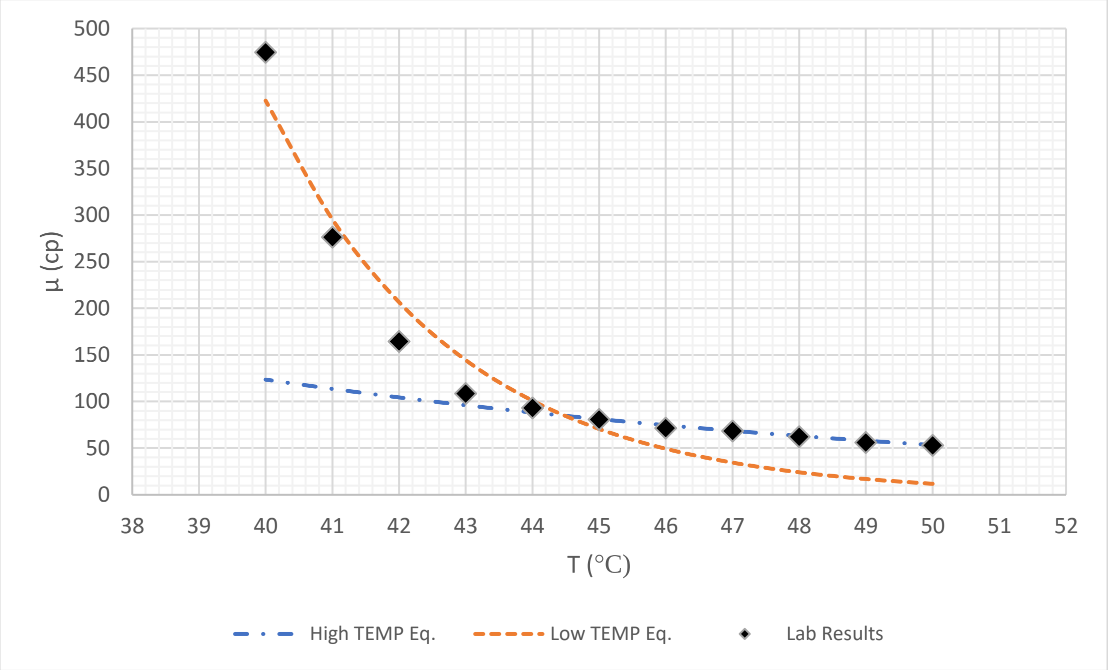

Figure 4. 18 Calculated viscosities result of crude oil with hexane 5% at shear rate 6/s

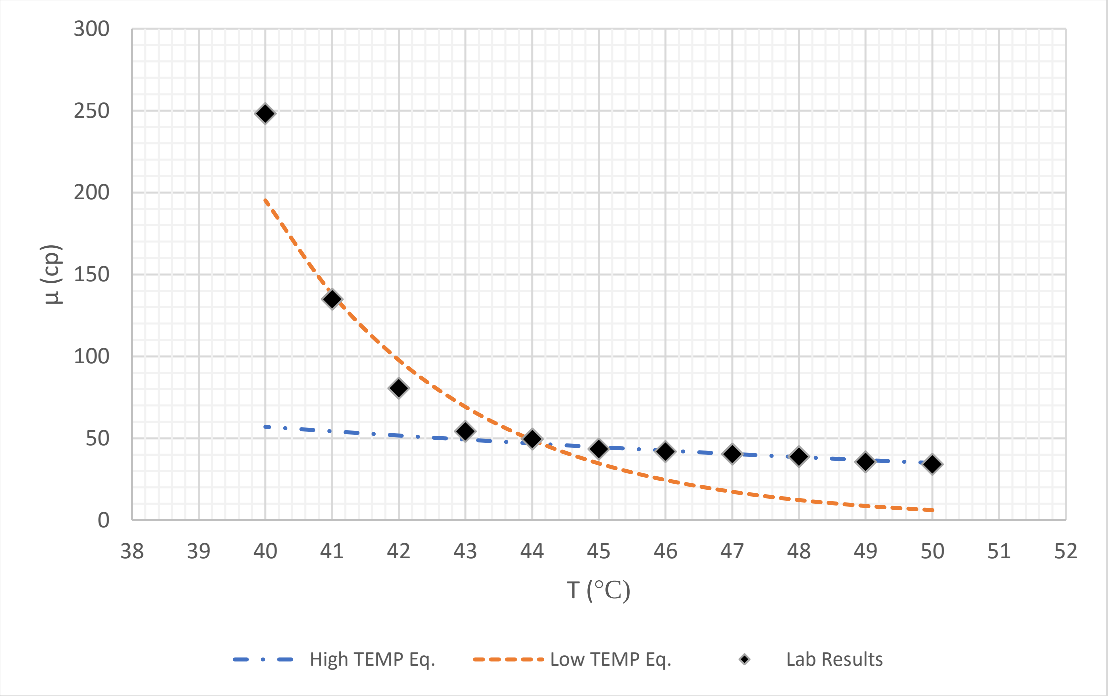

Figure 4. 19 Calculated viscosities result of crude oil with hexane 5% at shear rate 12/s

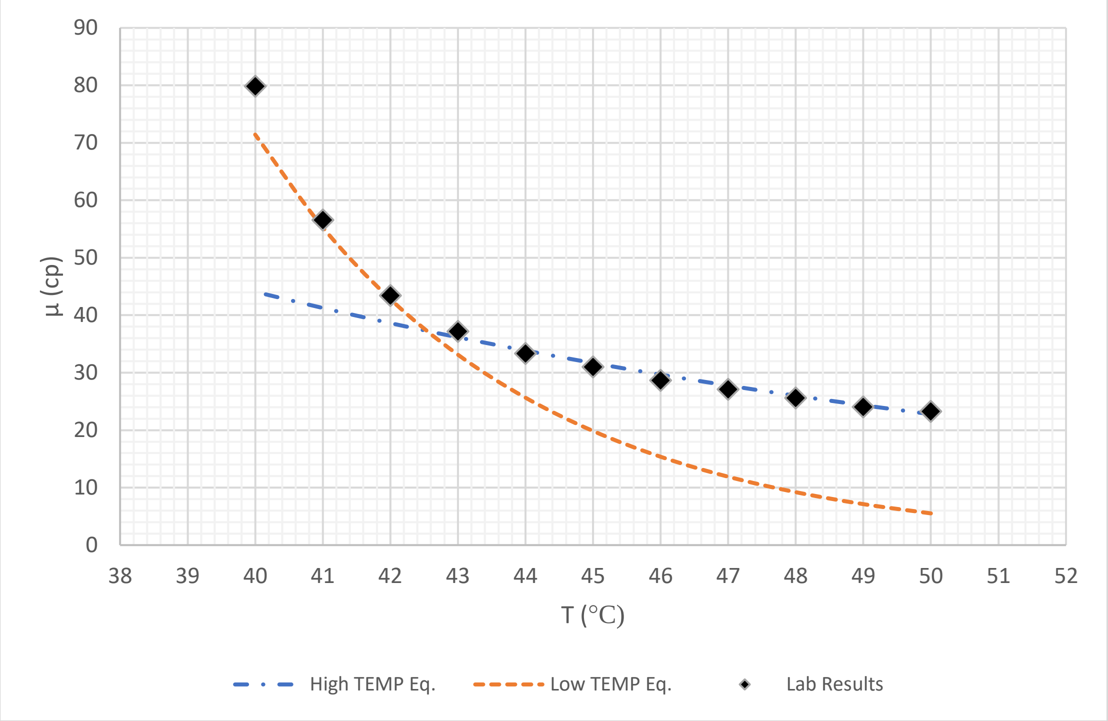

Figure 4. 20 Calculated viscosities result of crude oil with hexane 5% at shear rate 24/s

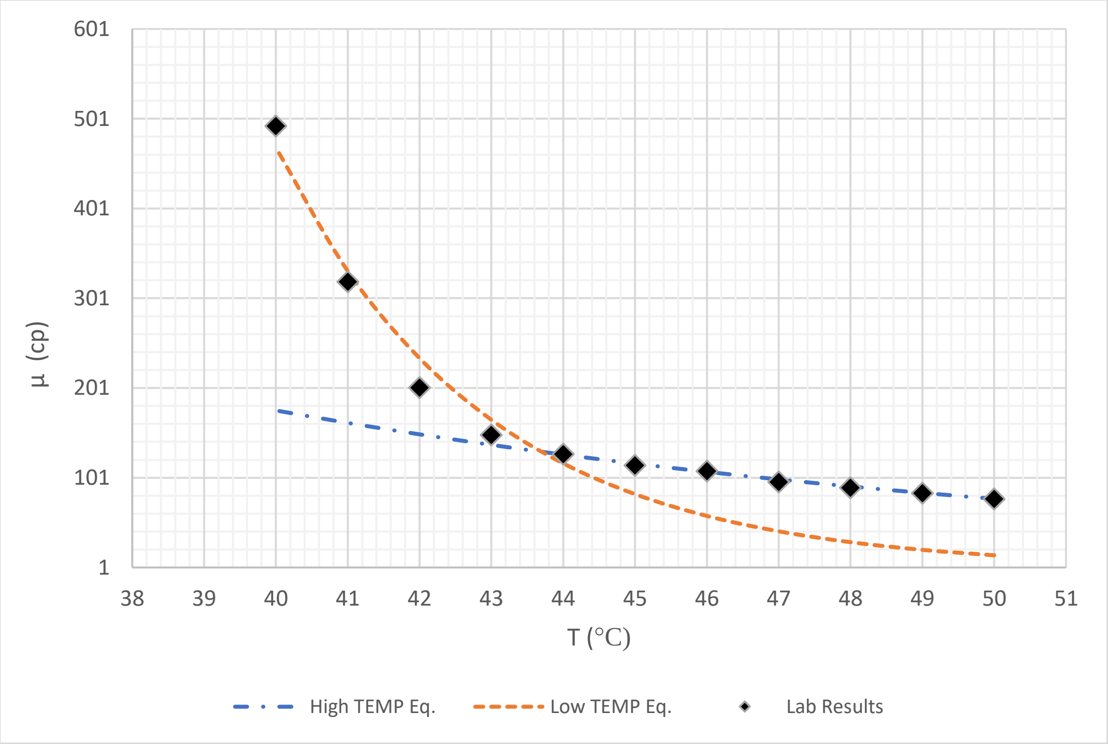

Figure 4. 21 Calculated viscosities result of crude oil with hexane 10% at shear rate 6/s

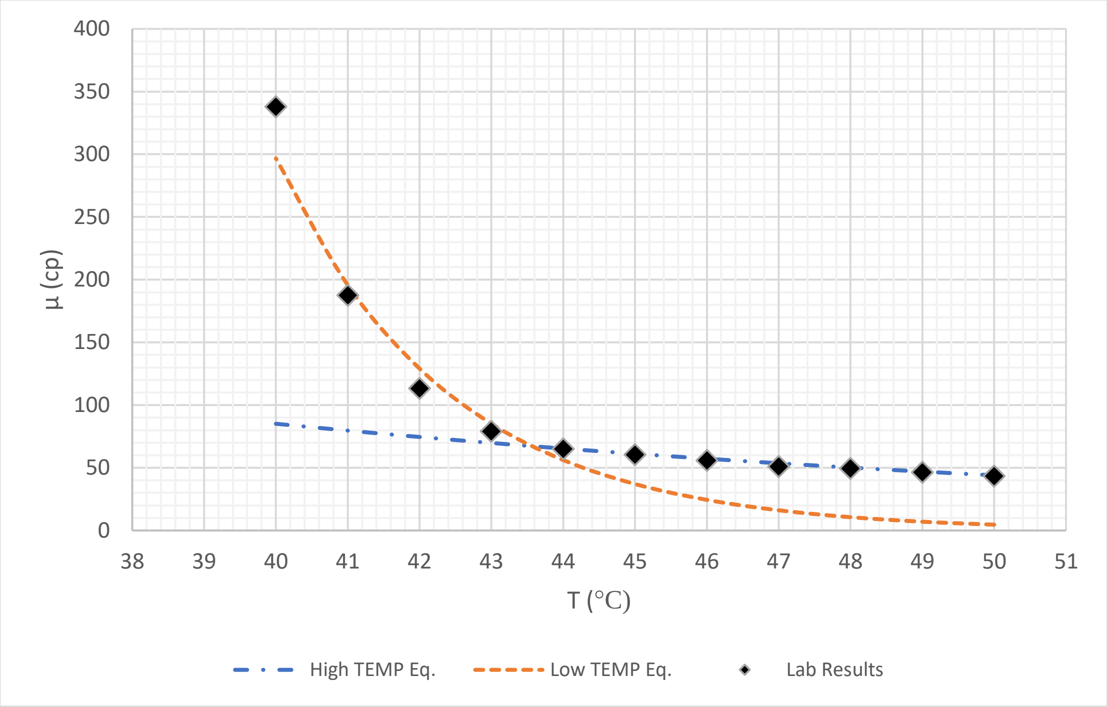

Figure 4. 22 Calculated viscosities result of crude oil with hexane 10% at shear rate 12/s

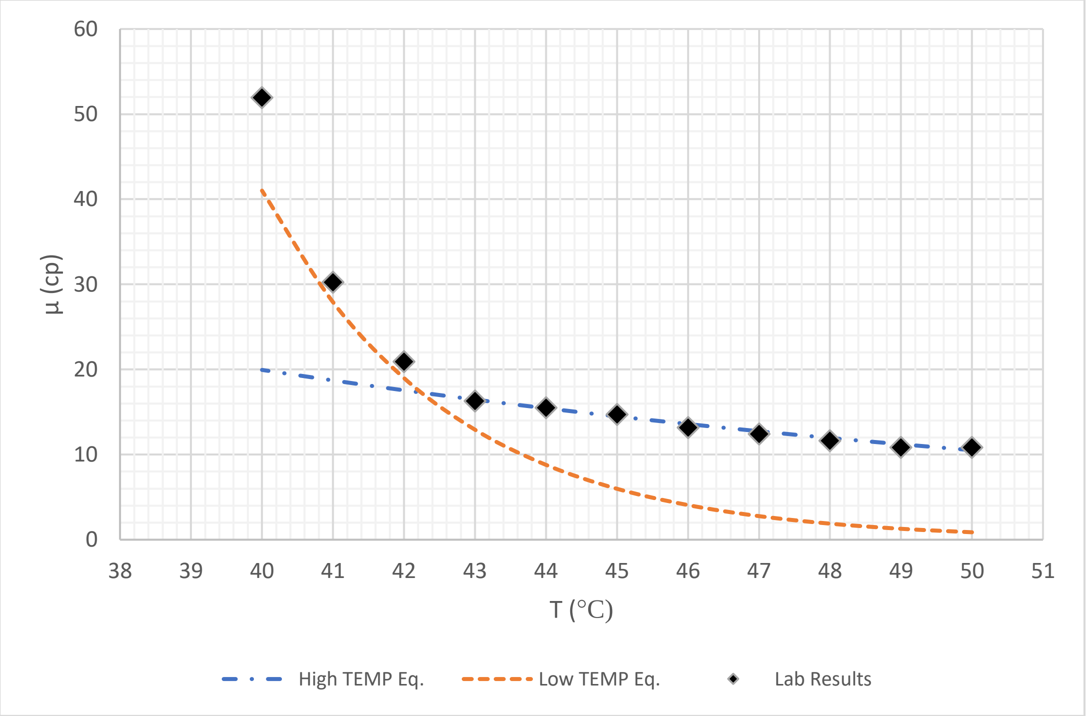

Figure 4. 23 Calculated viscosities result of crude oil with hexane 10% at shear rate 24/s

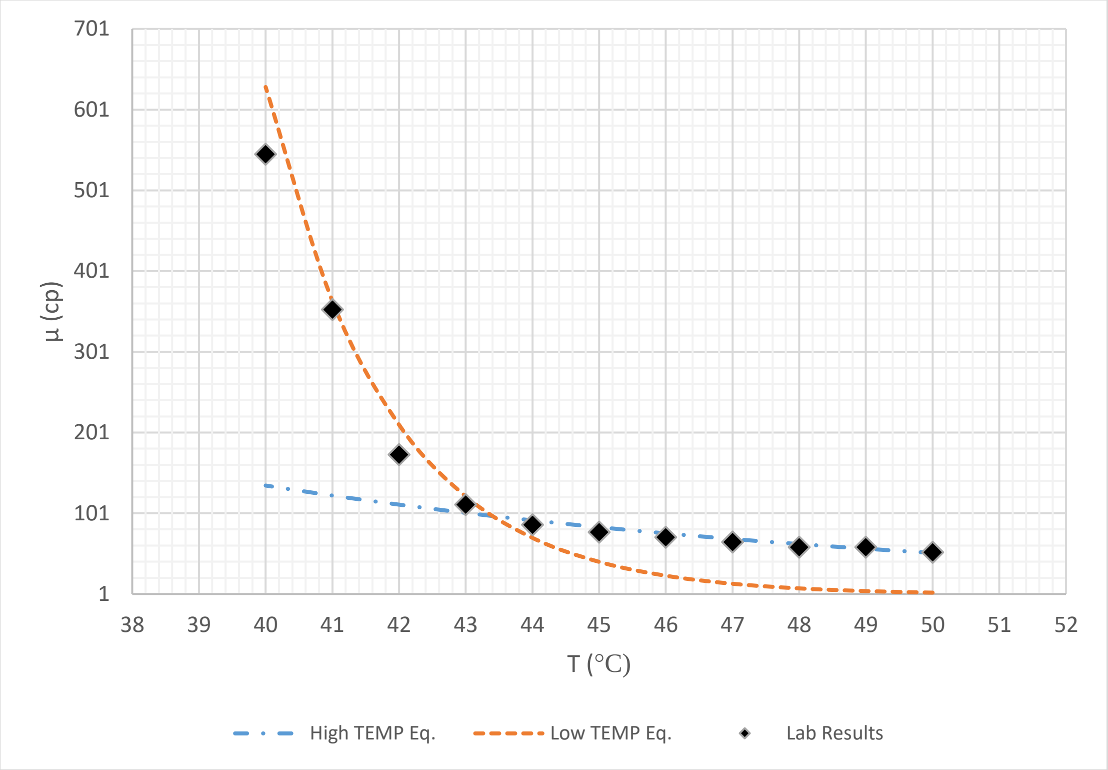

Figure 4. 24 Calculated viscosities result of crude oil with hexane 15% at shear rate 6/s

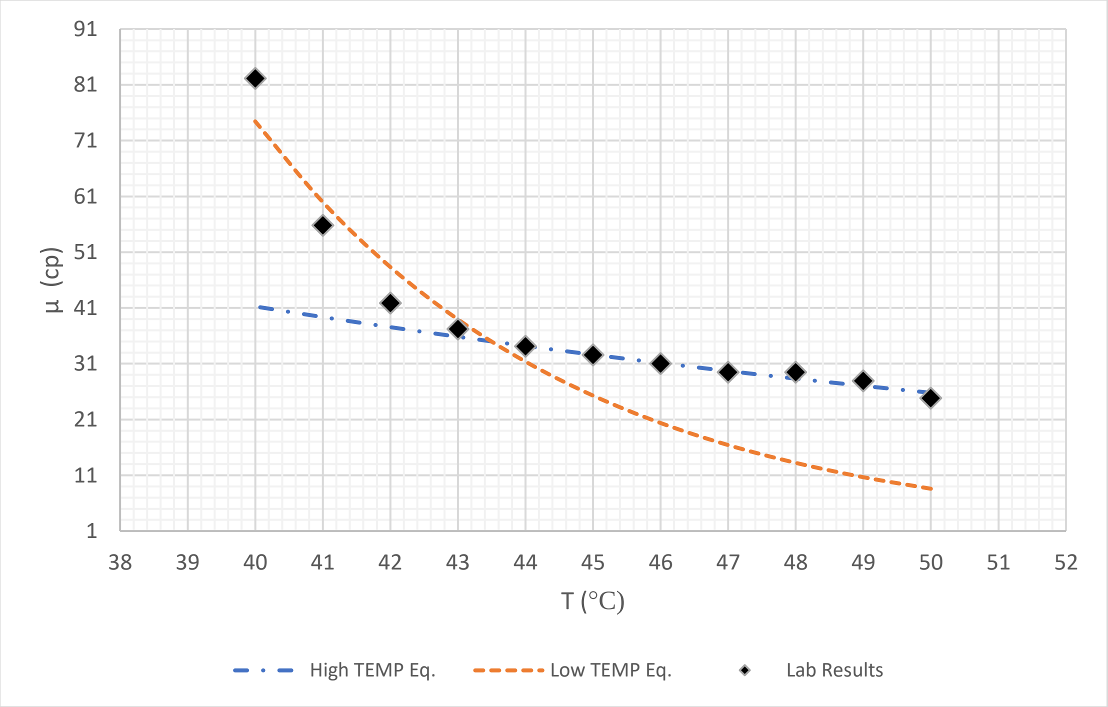

Figure 4. 25 Calculated viscosities result of crude oil with hexane 15% at shear rate 12/s

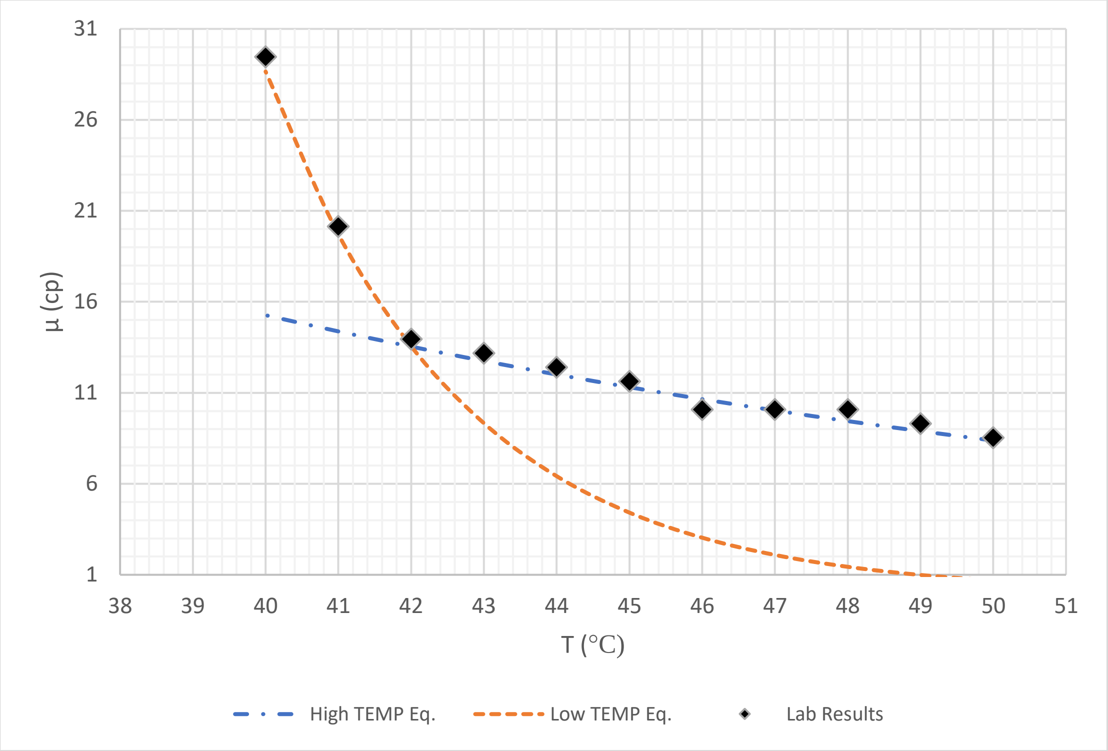

Figure 4. 26 Calculated viscosities result of crude oil with hexane 15% at shear rate 24/s

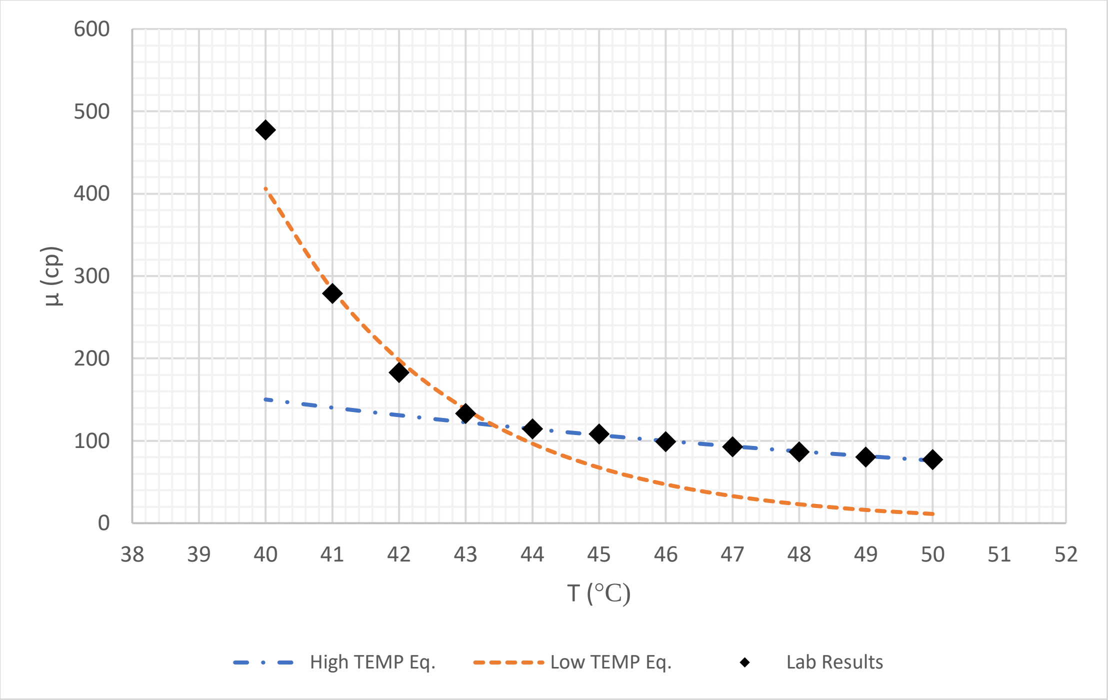

Figure 4. 27 Calculated viscosities result of crude oil with hexane 20% at shear rate 6/s

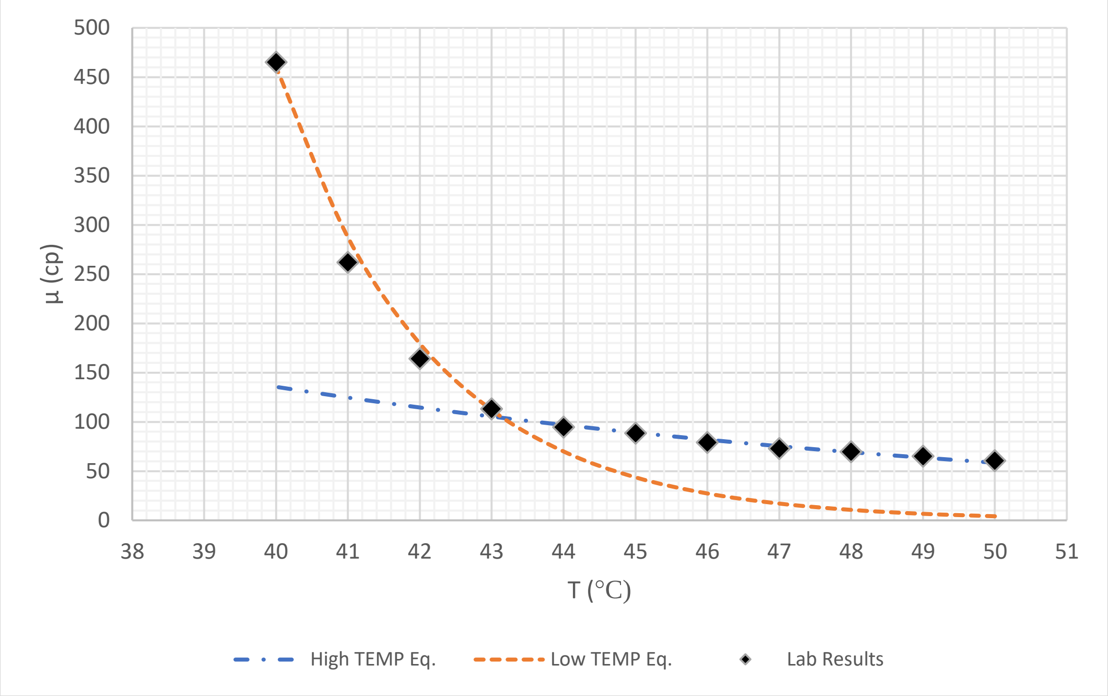

Figure 4. 28 Calculated viscosities result of crude oil with hexane 20% at shear rate 12/s

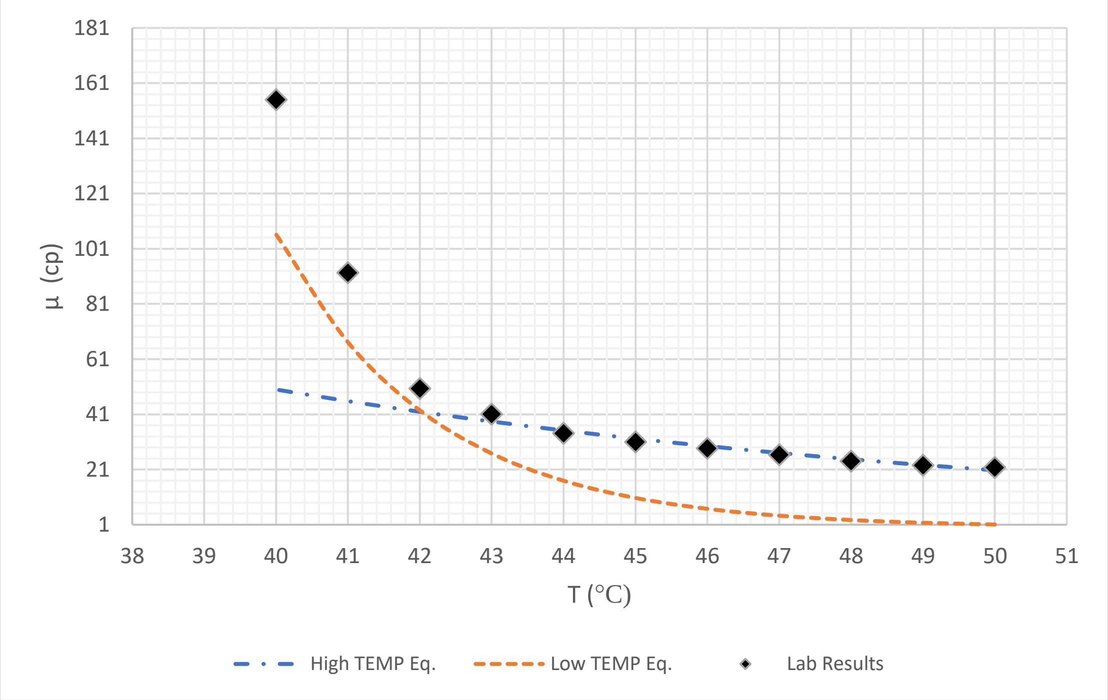

Figure 4. 29 Calculated viscosities result of crude oil with hexane 20% at shear rate 24/s

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