Investigation of crude oil emulsion stability

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved by,

_____

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
JANUARY 2015

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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Mehdi Nikfekr

ABSTRACT

Nowadays, crude oil production facing costly and serious problem due to produce crude oil along with water in form of an emulsion. Emulsion defined as mixture of two immiscible liquid such as oil and water by an emulsifier agent which are needed to be separated or treated by using separator tank and demulsfier to meet crude oil specification for buyer. Select and apply different demusfier in order to separate or treat a crude oil emulsion is costly operation also lead to waste a lots of time and required to specify emulsion stability to select specific demulsifiers to separate emulsions. The objective of this project is to evaluate the stability of synthetic water in oil emulsions. As well as to develop mathematical correlations to predict or determine emulsion stability.

In this project, four different methods are used in order to obtain crude oil emulsion stability. These methods consists of pH measurement, refractive index, dielectrical constant as well as stability measurement by using Turbiscan device. These standards methods investigate regarding droplet size of dispersion phase in crude oil emulsions as well as interfacial film that forms around water droplets which are the main factor to identifying crude oil emulsion stability.

The results obtained from pH measurement shows that as values of pH of crude oil emulsion increases, it leads to have weaker stability of crude oil emulsion, and this is because of interfacial film that formed by Asphaltenes are strongest but on the other hand, by increasing in pH it gets weaker. In refractive index measurement which is defined as speed of light through medium it investigate that as amount of water existence in emulsion are increases, the refractive index values decreases as it may detects more water droplets and as amount of water increases emulsions are less stable. Also, in Turbiscan methods, amount of sedimentation of water droplets in crude oil emulsions are measured as the values increases, it means less stable is.

After investigation of obtained results from experiments, also based on properties of crude oil emulsions samples it can be achieved crude oil emulsion stability correlations in order to predict stability of crude oil emulsions.

ACKNOWLEDGEMENT

I would like to express my gratitude and appreciation to all those who gave me the possibility to complete this report. A special thanks to my final year project supervisor Dr. Sulaimon Aliyu Adebyor, whose help, stimulating suggestions and encouragement, helped me to coordinate my project. Last but not least, to my beloved university Technologi Petronas for given me this opportunities to study here.

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CHAPTER 1

INTRODUCTION

1.1 Background

Emulsion thermodynamically considered as an unstable liquid and liquid system due to having tendency to separate and reduction its interfacial energy. Emulsions tend to show kinetic stability, therefore, it is vital to classify oil field produced emulsions based on kinetic stability and are categorized into three groups. First group includes loose emulsion which may be separated in few minutes. On the other group of kinetic stability involves Medium emulsion which will be separated in tens of minutes. Last group is known as tight emulsion and separate in hours or days. Crude oil emulsion stability it could be investigated based on their droplet size and interfacial films that form around water droplet which identify how stable is an emulsion. In this project all the factors that affecting stability are studied.

1.2 Problem statement:

Select and apply different demusfier in order to separate or treat a crude oil emulsion is costly operation also lead to waste a lots of time because each crude oil emulsions having different composition and property which leads to have different emulsion stability and specific emulsifiers need to be used in order to separate emulsions. Therefore, since emulsion stability will be different and the need apply different emulsifier to find out most suitable one is not economically efficient. As a result, by classifying emulsion stability range for crude oil emulsions it can be very useful and economically for production company.

1.3 Objective:

- To evaluate the stability of synthetic water in oil emulsions.

- To develop mathematical correlations to predict or determine emulsion stability.

1.4 Scope of study:

The scope of study is to measuring stability of different types of crude oil emulsion which consists of different API gravity and other properties to achieve my objectives which is to obtain a correlation or relationship that if by knowing characteristics of crude oil and water to be able to predict the stability of a crude oil emulsion range without do experiment.

CHAPTER 2

LITERATURE REVIEW

2.1 Stabilizing mechanism

According to (L.L. Schramm ed. 1992) the kinetic stability of water in oil emulsions is a result of droplet size of water as well as interfacial film that formed around water droplet by emulsifiers. There are four group of mechanisms including: phase inversion, coalescence, aggregation as well as sedimentation. Sedimentation defined as Falling water droplets of an emulsion occur due to difference between the oil and water density. Aggregation or flocculation refer to a grouping of water droplets emulsion which approach together and do not make difference in surface area. Coalescence is a critical mechanism which is describe as merger of water droplets dispersion to form a large single drop with reduction in surface area. Therefore, existence of emulsifiers lead to prevent these mentioned mechanism not to being happened otherwise it will break down the emulsion.

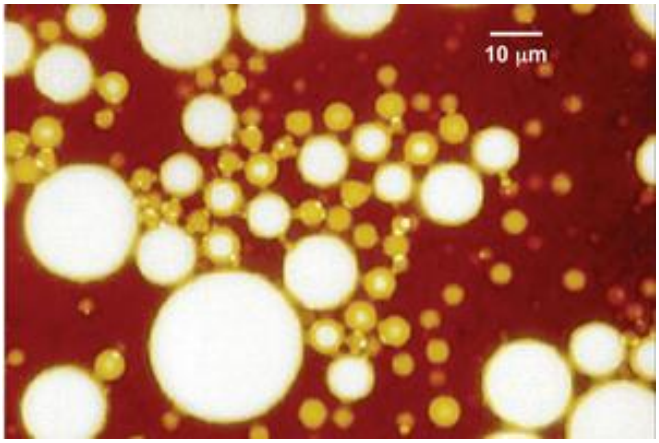

Figure 2.1 Interfacial film of an emulsion

2.2 Surface films and stability to coalescence:

On the research conducted (Jones, T.J., Neustadter, E.L., and Whittingham, K.P. 1978) The Surface film that form around water droplets in crude oil emulsions considered as one of the important criteria in emulsion stability. Another significant criteria in emulsion stability is existence of high molecular weight polar molecules which is essential in forming of surface film and this film by increasing the interfacial viscosity lead to enhance the emulsion stability.

There are several significant properties that interfacial film depends on are consists of:

- Temperature
- Type of crude oil such as asphaltic, paraffin
- Aging time
- Composition and pH of water

On the other research (Strassner, J.E. 1968) expressed that Emulsion stability has a critical correlation with incompressible interfacial film based on their mobility which consists of Rigid or solid films as well as Mobile or liquid film. Rigid film are categorized by high interfacial viscosities and are solid skin around water droplet which are soluble. Very fine solids leads to stabilize interfacial films and provide a barrier to water droplet coalescence and as result leads to enhance emulsion stability. However, liquid film are inverse in terms of characterization. Mobile films specified as low interfacial viscosity with less stability compared to solid films. Moreover, its formation is when an emulsion breaks (emulsifiers) is being added to a crude oil emulsion.

2.3 Factors affecting crude oil emulsion stability

After investigating interfacial film as a primary factor on emulsion stability, therefore is highly significant to consider the factors that affecting interfacial film (Strassner, J.E. 1968) which are characterized as bellows:

- Droplet size distribution
- Heavy polar fraction in crude oil
- pH of brine
- Temperature effect

- Solid materials consists of organic (Asphaltenes and waxes) and inorganic such as clays, scales and corrosion products.

There are three important emulsifier components which including: Resins, Asphaltenes and soluble organic acids which are concentrated in higher boiling polar fraction and main components of interfacial films around water droplet that tend to stability of an emulsion.

2.3.1 Asphaltenes

Generally Asphaltenes are component of crude oil with a complicated polyaromatic molecules and involves two unique phases consists of soluble and insoluble In benzene and in low molecular weight n-alkane respectively. In terms of color are dark brown.

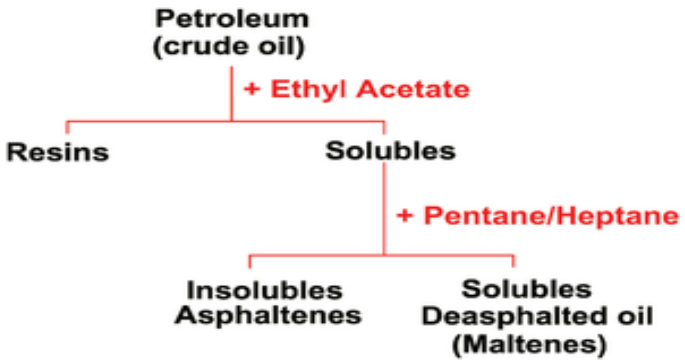

Figure 2.2 Classification of petroleum Asphaltenes.

By investigating Asphaltenes behaviors as well as its properties it can be find out its important effects on crude oil stability conditions. When Asphaltenes is existence in colloidal state it will increase in oil emulsion stability. It is important to mention that Asphaltenes itself stabilized by Resin moreover, there is a strong studies that indicates precipitated of Asphaltenes from crude oil leads to increase more properties stability of emulsion.

According to (Strassner, J.E. 1968) several experiences had be done regarding Asphaltenes that existence of Asphaltenes in crude oil emulsion make our emulsion strong enough which characterized by solid film. And removal of them tend to have loose emulsion formation of mobile films. Actually, presents of Asphaltenes can prevent these film to be drained and do not let drop to coalescence by aggregation around film and due to have colloidal stabilization. As a results based on (Bobra, M. 1990.) and (Strassner, J.E. 1968) there are two important criteria factors in controlling of emulsion stability which are consists of change in amount of Asphaltenes and Alkane in crude oil.

Resin has a high tendency to have combination with Asphaltenes which result to form micelle and responsibility for solid or mobile film formed, therefore indicate a relation to stability an emulsion.

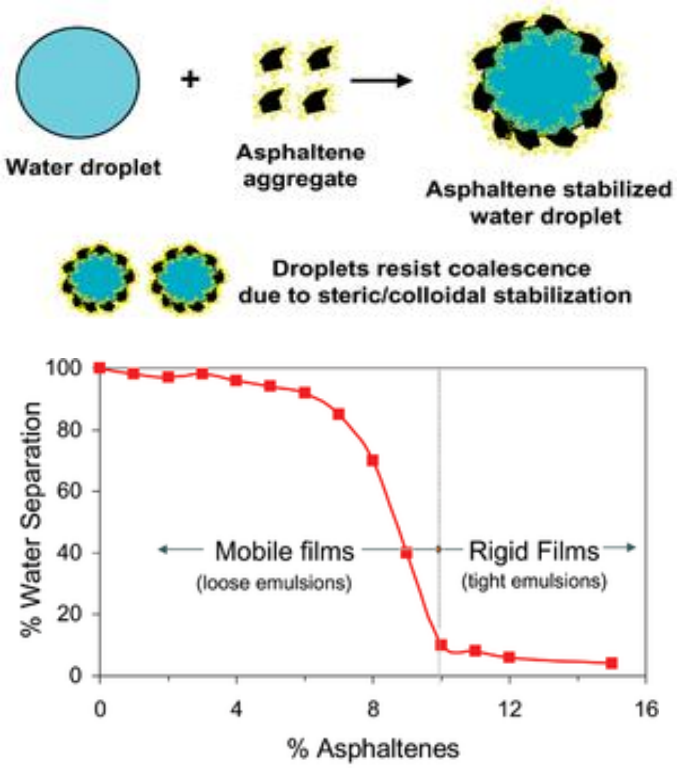

Figure 2.3 Effect of Asphaltenes

2.3.2 Solids particle:

According to (Tambe, D.E. and Sharma, M.M. 1993) Fine solid particles as an important factors have a significant role in emulsion stability.After diffusing into crude oil emulsion interface and form rigid film can stop or in other word preventing coalescence of droplet an emulsion. Moreover, fine solids are in terms of stability affect depending on these following agents:-

- Solid particle size
- Antiparticle interactions
- Wettability of the solids

2.3.3 Temperature effect

In general, any change in temperature can lead to change in systems and affecting physical properties of systems such as oil and water, solid or liquid films and as result change in our emulsion stability. However, temperature put its effect in the viscosity. Therefore, there is an inverse relation between temperature and viscosities. Also, it has proportional relation with stability. While temperature of system increase viscosity decrease as well as stability decrease (Jones, T.J., Neustadter, E.L., and Whittingham, K.P. 1978).

2.3.4 Droplet size

Stability of crude emulsion investigation based on Droplet size distribution refers to size of droplet. If droplet is smaller means that is more stabilized. Also, there is a good correlation between time and size of droplet. Whatever, our droplet size is smaller it needed greater time to being separated.

2.3.5 pH of water

According to (Kimbler, O.K., R.L. Reed, A., and Silberberg, I.H. 1966.) It is important take into consideration that PH of water act as one of the main factor in stability of oil emulsion. According to straassner expressed the relationship between Asphaltenes and PH of water and its effect on interfacial film. He indicated that if interfacial film formed by Asphaltenes are strongest but on the other hand, by increasing in PH it gets weaker. Then, amount of PH has influence on type of emulsion that formed, low PH produce water in oil emulsion. Whereas, High PH produces oil in water emulsions.

2.3.6 Dielectric constant

Dielectric constant representing the ability of materials to store the electromagnetic energy as it passes through (J.P Robinson et al., 2012). The dielectric constant it can be used to characterize crude oil emulsions. Any change in DC (Dielectric constant) demulsifier dosage it could use as measuring crude oil emulsion stability. This method are used for ranking, screening as well as selecting demulsifier resolution.

2.3.7 Refractive index

The first laboratory instrument to accurately measure the refractive index of liquids was developed by Ernst Abbe in 1874. Refractive index measurement is actually a measurement of the speed of light in a medium. In other media the speed of light is lower than this value, and the refractive index, R.I., of a medium is a measure of how much the speed of light is reduced in the medium. The refractive index (n) of a medium is defined as the ratio of speed of light in vacuum (c) in to that in the medium (v):

$N = C/V$ …… (2.1)

The speed of light in a medium depends on the medium itself, temperature and wavelength .In crude oil emulsion as amount of water which dispersed in crude oil increases the refractive index value decrease and this is due to detect the too much water droplet which is available is sample

CHAPTER 3

METHODOLOGY

This experiment conducted based on four different methods for investigating crude oil emulsion stability and characterize the factors affecting stability. These methods are consists of Refractive index method, pH measurement, Dielectric constant as well as stability measurement using Turbican device.

3.1 Sample preparation

This experiment conducted based on three different crude oil samples which are ordered from PETRONAS refinery located in Melaka. Each crude oil consists of different properties such as API gravity, viscosity as well as density which are measured in laboratory separately. Density of each crude oil is measured by density meter at room temperature.

Figure 3.1 Density meter

Following table display crude oil samples properties:

Table 3.1 Crude oil properties

Properties / crude oil	**Crude oil A**	**Crude oil B**	**Crude oil C**
DENSITY (g/mL)	0.9079	0.940	0.946
API GRAVITY(°API)	24.35	19.03	18.07
VISCOSITY (Cp)	600.99	758.4	404.08
Color	Black	black	Light Brown

After specifying crude oil samples properties, this experiment starts with preparing crude oil emulsions, which are done based on two different ratio 30%/70% and 50%/50% water in oil emulsions. In order to prepare the Brine of sample, 2 gram of NaCl added to100 ml of distilled water and by using stirrer magnetic machine are mixed for 20 minutes or as long as the salt dissolved in water to form a brine.

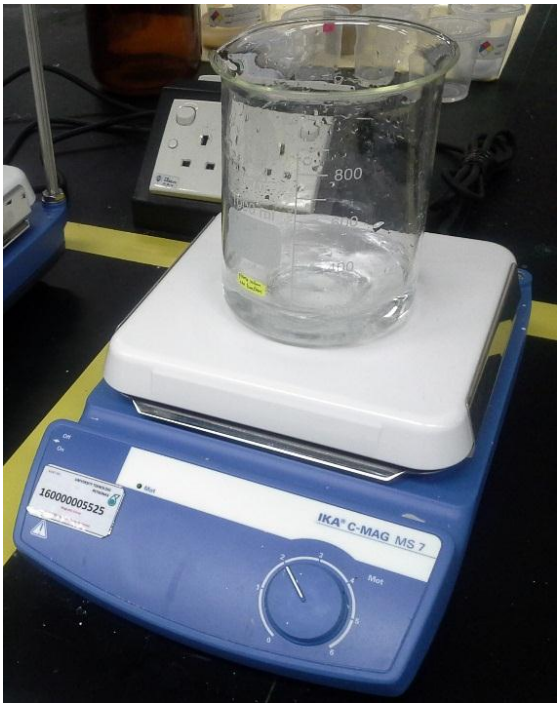

Figure 3.2 Stirrer magnetic

At this stage, by specifying the amount of crude oil and brine to make an emulsion, they are mixed for exactly 5 minutes to form an emulsion by using constant speed mixer.

Figure 3.3 Constant speed mixer.

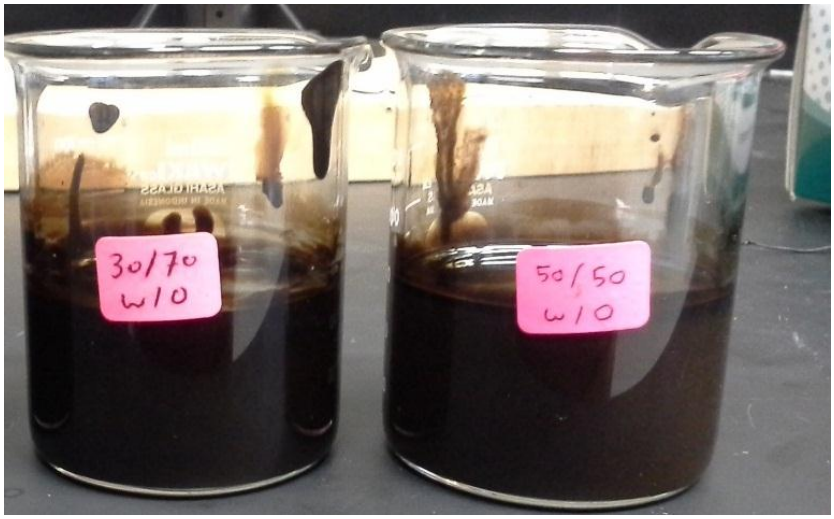

Figure 3.4 Crude oil emulsions.

3.2 Sample testing

After preparing 30/70% and 50 /50% crude oil emulsion, then proceed to measuring four different method characterizing crude oil emulsion which consists of PH method, Refractive Index, Dielectric constant as well as stability measurement using Turbiscan device at three different temperature such as (25, 50, 70) **°C.**

3.2.1 pH meter

The pH of all of the crude oil emulsion samples and crude oil itself are measured separately by using PH meter at room temperature to investigate the pH of samples. *Results are shown in result and discussion section.

Figure 3.5 pH meter

3.2.2 Refractometer

Second method is to measure refractive index of crude oil emulsion by using refractometer at (25, 50, 70) **°C to** find out light reflection behaviour on crude oil emulsion stability.

Figure 3.6 Refractometer

2.2.3 Dielectric constant method

This method by using a probe which located in middle of samples and a network analyzer which transfer frequency through probe into crude oil and measurement are shown on Monitor. This measurement are done based on mentioned temperatures.

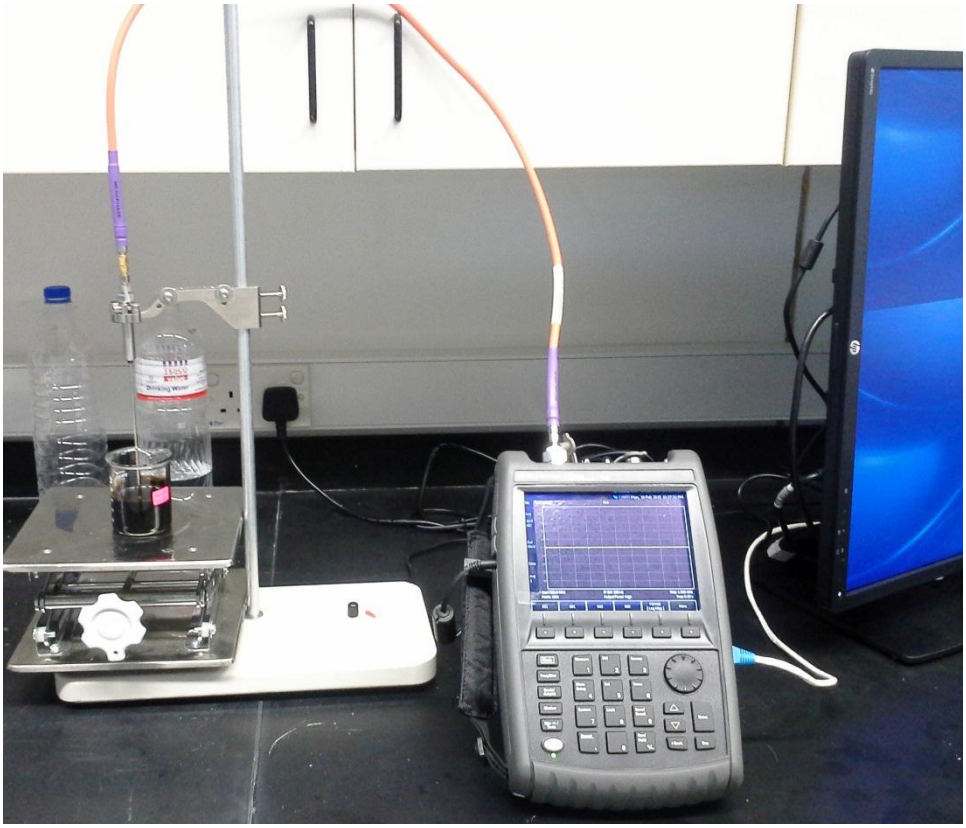

Figure 3.7 Network Analyzer.

2.2.4 Turbiscan classic device

This measurement is considered as a critical methods to investigate stability of crude oil emulsion by scattering particle droplets in dispersion phase which is brine. By analyzing the amount of percentage of sedimentation of droplets in different times, it can be find out the stability of crude oil emulsions as amount of sedimentation percentage increase ,the stability decrease due to settle down the particle droplet od dispersion phase.

Figure 3.8 Turbicsan classic

CHAPTER 4

RESULTS AND DISCUSSION

In this section all obtained results from experiment are given for a3 different crude oil emulsions for different ratios of 30:70/ and 50:50 at different temperature in the detail and analysis are done accordingly.

4.1 pH results and analysis

Following table indicates the results that are obtained from pH measurement.

Table 4.1 pH results

Water oil ratio	pH values of crude oils			
	A	B	C	Brine
30:70	7.05	7.20	8.30	6.64
50:50	6.60	7.02	8.708	6.64
0:100	6.72	7.12	8.955	6.64

As it discussed earlier pH has significant effect on stability of crude oil emulsions. According to (straassner) expressed the relationship between Asphaltenes and PH of water and its effect on interfacial film. interfacial film formed by Asphaltenes are strongest but on the other hand, by increasing in PH it gets weaker From above data it can be analyzed that crude oil emulsion A having PH is range of 6 and is having lower PH compare to other two crude oil emulsions.

Because the interfacial film formed by Asphaltenes are more stronger bond and take more time to be destabilized or getting weak and as the ration of crude oil emulsion changed from 30%70% to 50%50%, also the amount of PH ratio increase and leads to having weaker stability. Furthermore, crude oil emulsion B having PH in range of 7 and compare to crude oil emulsion C is more stable since the amount of PH is increasing in crude oil emulsion C to range of more than 8. It can be concluded that by increasing in amount of PH and effect on interfacial films that leads the crude oil emulsion become less stable.

4.2 Stability measurement results and analysis

It is essential to investigate that how stable is an emulsion which in this method by using turbiscan device the measurement is done, based on back scattering profile and observing the percentages of sedimentation of each crude oil emulsion samples.

The sedimentation phenomenon can be observed with emulsion and suspension when the density of the dispersed phase is higher than the density of the continuous phase. It can also occur after a flocculation or a coalescence when the size of the particles is too big. This instability will eventually lead to a phase separation. After conducting the test following table indicate the results of sedimentation that obtained at different temperatures.

Table 4.2 Stability results

Crude	**Stability (sedimentation) 30% 70% W/O**			**Stability (sedimentation) 50% 50% W/O**		
Temperature	25 °C	50 °C	70 °C	25 °C	50 °C	70 °C
Crude A	0.0092	0.038	0.057	0.00598	0.0534	0.132
Crude B	0.0189	0.05	0.072	0.00806	0.01412	0.0192
Crude C	0.037	0.122	0.143	0.0106	0.368	0.421

From above table, that as the amount of sedimentation increase it indicates that more dispersed particle droplet is going to settle down and it is showed faster separation or in other hand, lesser crude oil emulsion stability. According to above table, the crude oil emulsion A having less sedimentation percentage as a result more stable crude oil emulsion and due to effect of temperature on crude oil emulsion as it is increased leads to less stability, however, in this experiment the result of temperature is obvious and as temperature from 25 degree reached to 70 degree is caused to have less stable crude oil emulsion. It can be concluded that Crude oil emulsion A more stable compare to B and C. Crude oil emulsion C having weakest stability. And this result are work for both crude oil emulsion ratios.

4.3 Refractive index results

Refractive index or known as (RI) is defined as speed of light go through a medium and reflected to directions. Here I would like to discuss the behavior of RI in crude oil emulsions as the ratio and temperatures of crude oil emulsion changes and relate it to stability of crude oil emulsion stability. Below are RI results that achieved during experiment:

Table 4.3 Refractive index results

Crude	**Refractive index 30% 70% W/O**			**Refractive index 50% 50% W/O**		
Temperature	25 °C	50 °C	70 °C	25 °C	50 °C	70 °C
Crude A	1.5116	1.5012	1.492	1.49	1.4835	1.4759
Crude B	1.5449	1.5329	1.5263	1.5384	1.5257	1.5154
Crude C	1.573	1.5028	1.4271	1.5168	1.4334	1.3603

Every crude oil emulsion are plotted as bellows:

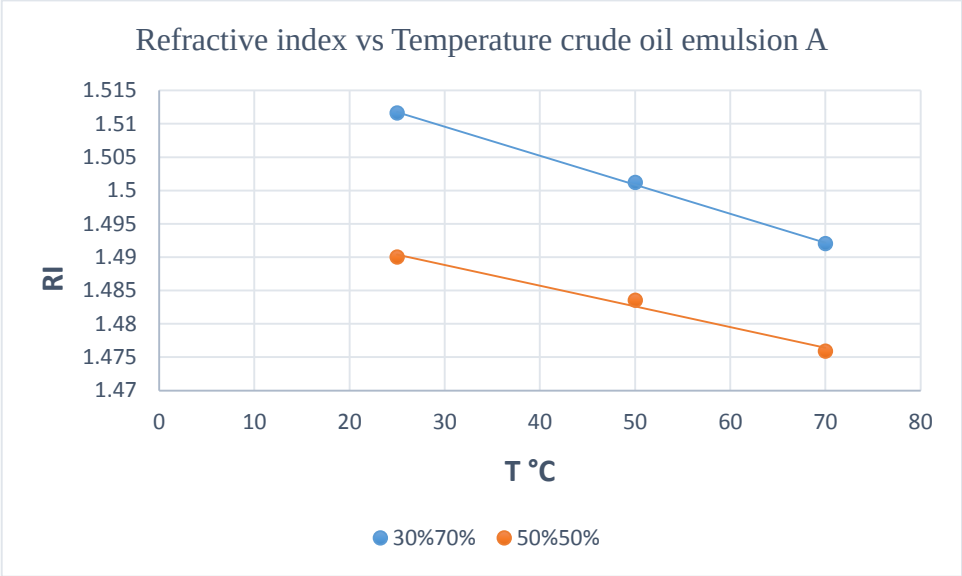

Figure 4.1 30:70 / 50:50 for crude oil emulsion A

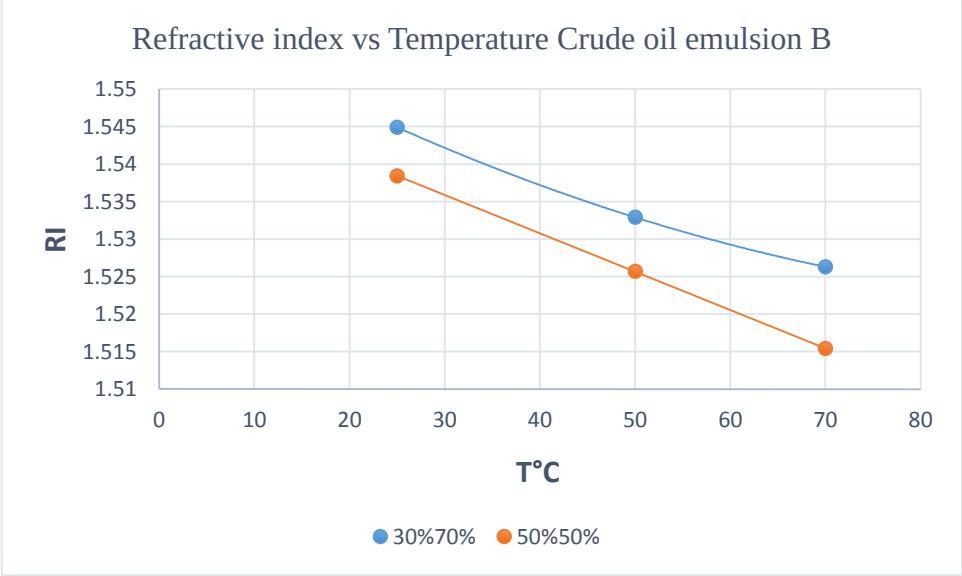

Figure 4.2 30:70 / 50:50 for crude oil emulsion B

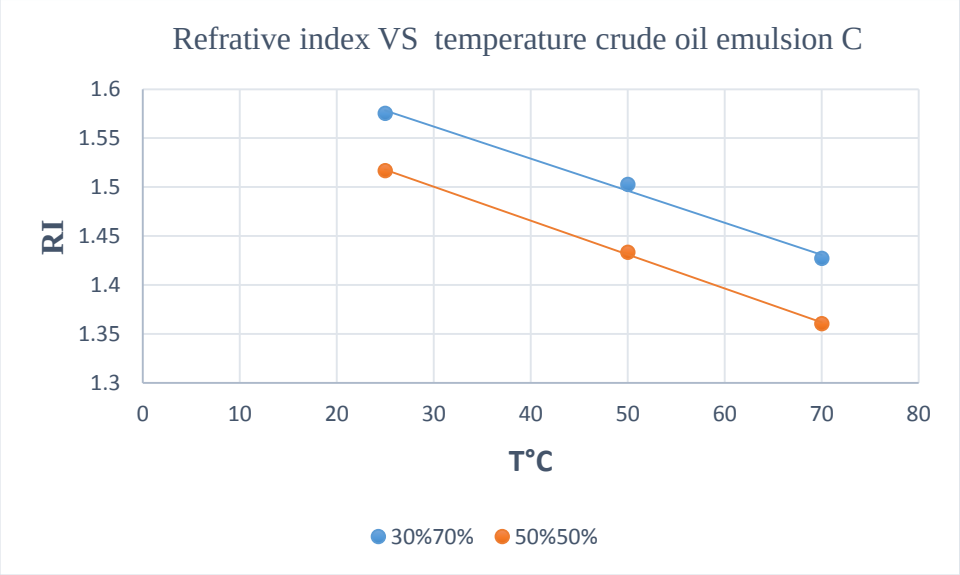

Figure 4.3 30:70 / 50:50 for crude oil emulsion C

4.2.1 Refractive index results Analysis

From definition of RI which is defined as the speed of light over that specific medium it can analyzed that as amount of water increase in crude oil emulsion, since the dispersion phase is water, RI getting lower values. This is clear by comparing ratios of 30%/70% and 50%/50%. Ratios of crude oil emulsions. When the water ratio is more in crude oil emulsions it means more particle droplet are available is emulsion that cause more difficulties for light to pass through samples and detect water droplet in crude oil emulsions and indicates that sample is water in oil emulsions and obviously as the percentages of water increases is leaded to have weaker crude oil emulsions on the other hand , the effect of temperatures on crude oil emulsion is clearly shows that by increasing in temperature it leads to decrease in refractive index values.

4.3 Dielectric constant results

Tables shows results after dielectric constant measurement

Table 4.4 Results of dielectric constant

Crude	Dielectric Constant 30% 70% W/O			Dielectric Constant 50% 50% W/O		
Temperature	25 °C	50 °C	70 °C	25 °C	50 °C	70 °C
Crude A	84.05	64.40	63.15	26.73	25.06	46.98
Crude B	205.85	59.46	35.28	43.66	178.66	220.44
Crude C	232.76	260.2	190	201.72	182.1	280.14

Plotting the values of dielectric constant versus temperature, are showed as bellows:

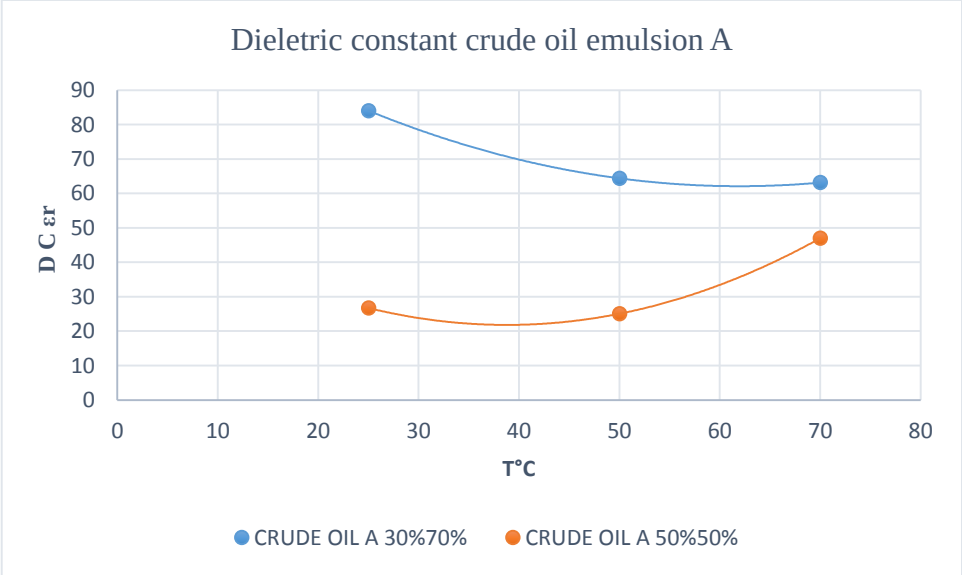

Figure 4.4 30:70/ 50:50 for crude oil emulsion A

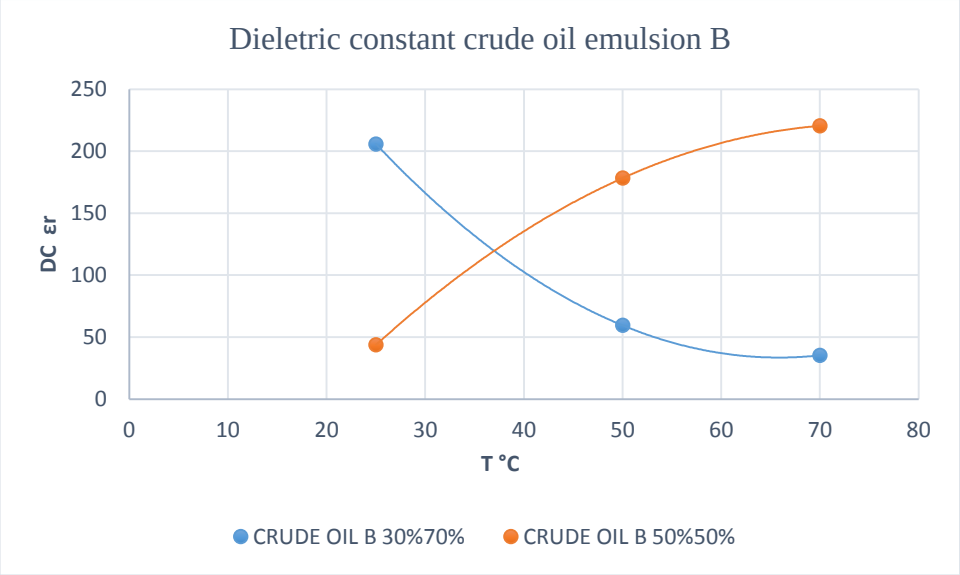

Figure 4.5 30:70/ 50:50 for crude oil B

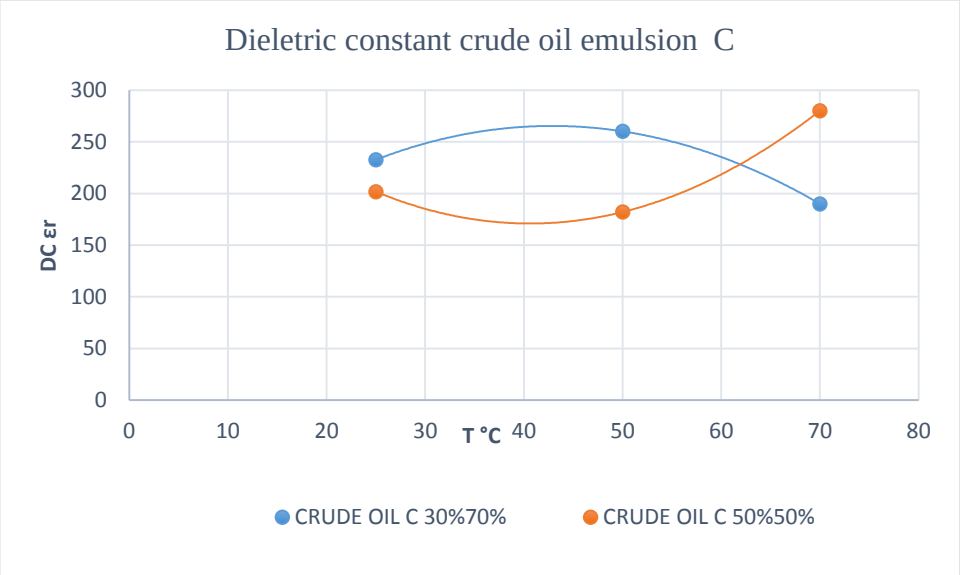

Figure 4.6 30:70/ 50:50 for crude oil C

Dielectric constant of crude oil emulsion increases with increasing water volume fraction in emulsion which can be explained according to Weiner equation knowing that the value of dielectric constant of water is much higher than oil. Based on this statement from the graphs it can be investigated that for ratio 50%50% the Dielectric constant having higher value compare to 30%70% W/O due to higher fraction of water in oil and higher conductivity of water also

which probe can detect more water droplet, DC value increase through the cycle up 70 ‘C, but on other hand, but in ratio of 30%70% W/O the dielectric constant value keep decreasing, this might be due to lack of water droplet at higher temperature which leads that DC probe cannot detect much droplet size which may be due to phase separation of crude oil emulsion.

4.4 Sensitivity analysis

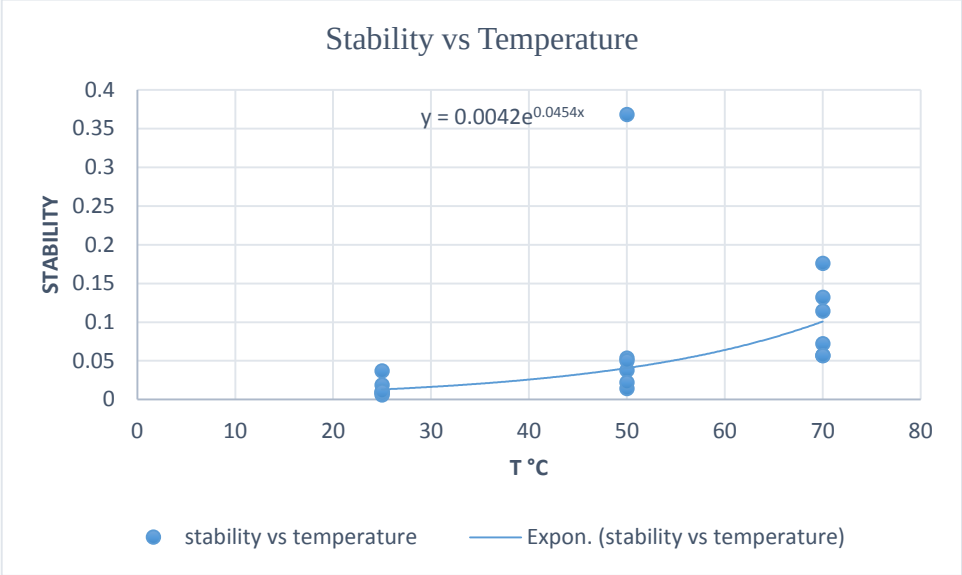

Figure 4.7 Stability vs temperature

This measurement is done to investigate the change of stability as temperature increases. From given graph is obvious that as temperature increases, stability increases, because change in systems and affecting physical properties of systems such as oil and water, solid or liquid films and as result change in our emulsion stability.

4.5 Stability correlation

Objective of this study is find the stability correlation that by applying crude oil properties into correlation it can estimate stability ranges. After collecting all properties could achieve correlation.

By having properties of crude oil as input data and also stability as output data following correlation achieved.

Correlation is as follow:

$$Y = \exp(a*X1+b*X2+c*X+d*X4+e) \quad \text{..... (4.1)}$$

Where

Y= stability (in this case sedimentation)

a= - 0.00662

b = 0.0297

c= -0.0002707

d= 4.91

e= -2.648

CHAPTER 5

CONCLUSION AND RECOMMENDATION

Crude oil production facing costly and serious problem due to produce crude oil along with water in form of an emulsion which need to be separated.so, it is needed to identify crude oil emulsion stability in order to choose best demulsifier for separation water from oil. . After characterizing my three crude oil emulsion samples in different temperatures all observation and interpretations are explained. Four different methods are used. Such as refractive index, dielectric constant, PH measurement as well as stability measurement.

-The purpose of this project is to investigate the crude oil emulsions stability by using different methods to find out the factors affecting on emulsion stability.

- To evaluate the stability of synthetic water in oil emulsions.

- To develop mathematical correlations to predict or determine emulsion stability.

- It can be recommend for this project that as number samples increases in leads to have more accurate results.

- In order to achieve better results it recommends to determine chemical properties of each crude oil samples.

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