

**THE EFFECT OF INORGANIC SOLID PARTICLES ON WATER AND
CRUDE OIL EMULSION STABILITY**

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ABSTRACT

Emulsion either water-in-oil or oil-in-water emulsion can be important in almost all stages of upstream activities in petroleum industry nowadays. Crude oil have become an important sources of hydrocarbons in every part of the world which the formation of crude oil emulsion due to high paraffinic compound existed in the crude oil problem in dewatering stage of the crude emulsion. Many studies have been conducted in which to find a source that can stabilize the crude emulsion and how it affected the crude emulsion itself. Emulsion stability is determined by the extend of water resolved in which it is a measurement of the degree of disperse-phase coalescence after the separation process. All particles used were hydrophilic, can stabilized oil-in-water emulsions that are commonly found in refinery emulsions. In order to get better understanding in the effect of inorganic solid particles on the stability of crude oil emulsion, three types of crude oil emulsions were used and three types of inorganic solid particles was studied under various ratios of water/oil and weight percentage of the inorganic solid particles to the emulsions. The result showed that in increasing the weight percentage of the inorganic solid particle in the crude oil, the percent of water resolved is decreased slightly. And it was also observed that Kaolin has the largest stabilization effect in emulsions rather than Iron Oxide and Calcium Hydroxide. Comparing the crude oil used for this study, it is found that Masila crude oil is the most stable crude oil while Dubai and Miri crude oil show less stable properties. This is mainly because of the asphaltene content in the crude oil itself that can affect the stability of water and crude oil emulsion.

ABSTRAK

Emulsi samada air di dalam minyak atau minyak di dalam air merupakan factor yang penting dalam semua peringkat di dalam industri petroleum pada hari ini. Minyak mentah telah menjadi satu punca hidrokarbon yang penting di semua bahagian dunia di mana pembentukan emulsi minyak mentah disebabkan kandungan sebatian paraffin yang wujud di dalam minyak mentah adalah tinggi dan ini memberi masalah kepada proses membuang air dari dalam minyak mentah. Banyak kajian telah dijalankan di mana pencarian punca yang boleh menstabilkan emulsi minyak mentah dan bagaimana ia memberi kesan kepada emulsi minyak mentah itu sendiri. Kestabilan emulsi boleh ditentukan dengan mencari sebanyak mana air telah disingkirkan daripada emulsi kerana ianya merupakan darjah pengiraan penggabungan di antara minyak dan air selepas proses pengasingan selesai. Semua partikel yang digunakan adalah hydrophilic, di mana ia boleh menstabilkan emulsi air di dalam minyak yang biasanya terdapat di dalam emulsi penapisan minyak mentah. Untuk mendapatkan pemahaman yang lebih mendalam berkaitan kesan partikel tidak organik ke atas kestabilan minyak mentah, tiga jenis emulsi minyak mentah telah digunakan dan tiga jenis partikel tidak organik telah dikaji di bawah pelbagai nisbah di antara minyak dan kepelbagaian peratus berat partikel tidak organik ke atas emulsi. Keputusan kajian menunjukkan peningkatan peratus berat partikel tidak organik di dalam emulsi minyak mentah menyebabkan peratus air yang terasing daripada minyak menurun. Dan ianya juga telah dikaji yang Kaolin memberikan darjah kestabilan yang tinggi kepada emulsi berbanding Ferum Oksida dan juga Kalsium Hidroksida. Minyak mentah dari Masila merupakan minyak mentah yang paling stabil diantara minyak mentah Dubai dan minyak mentah Miri. Ianya kerana kandungan aspaltin yang tinggi di dalam minyak itu sendiri yang boleh memberi kesan kepada emulsi minyak mentah dan air.

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CHAPTER 1

INTRODUCTION

1.1 Background

Effective separation of water-in-crude oil emulsions is a central challenge for the oil industry all around the world, especially with the future increase in subsea and even down-hole processing of well-fluids. The mechanisms and properties governing emulsion stability are far from fully understood, but the indigenous surface-active crude oil components are believed to play a major role.

So, the basic knowledge here is to study and investigate the convenient way in separating crude oil and water emulsion. And as for that, we will look through the effect of inorganic solid particle on crude oil and water emulsion stability by experimenting and testing the crude oil with different kind of chemical reagents.

1.1.2 Chemical composition of crude oils

Even though crude oils are a continuum of tens of thousands of different hydrocarbon molecules, the proportions of the elements in crude oils vary over fairly narrow limits. Nevertheless, a wide variation in properties is found from the lightest crude oils to the highly asphaltenic crudes. The carbon content normally is in the range 83-87%, and the hydrogen content varies between 10 and 14%. In addition,

varying small amounts of nitrogen, oxygen, sulfur and metals (Ni and V) are found in crude oils.

Due to the complex composition of crude oils, characterisation by the individual molecular types is not possible, and elemental analysis is unattractive because it gives only limited information about the constitution of petroleum due to the constancy of elemental composition. Instead, hydrocarbon group type analysis is commonly employed. Knowledge of the distribution of major structural classes of hydrocarbons in crude oils is needed in various fields in the petroleum industry. Examples are studies related to reservoir evaluation, migration and maturity, degradation processes, processing, and environmental effects.

1.1.3 Emulsion

The concept of emulsions has been defined by IUPAC (1972) as:

“An emulsion is a dispersion of droplets of one liquid in another one with which it is incompletely miscible. In emulsions the droplets often exceed the usual limits for colloids in size.”

For the petroleum industry the usual emulsions encountered are water droplets dispersed in the oil phase (W/O), although the reverse situation is also possible. In addition to the usual emulsion types, multiple emulsions of for instance oil droplets dispersed in water droplets that are in turn dispersed in a continuous oil phase (O/W/O) can occur. Water droplets in crude oil emulsions might be up to 100 micrometers in diameter, which is large compared to the common definition of the upper limit of colloidal size (1 μm). The emulsion formation is a result of the co-production of water from the oil reservoir. During processing, pressure gradients over chokes and valves introduce sufficiently high mechanical energy input (shear forces) to disperse water as droplets in the oil phase.

All emulsions, perhaps with the exception of microemulsions, are thermodynamically unstable. However, the destabilization may take considerable time, and a stable emulsion is unable to resolve itself in a defined time period without some form of mechanical or chemical treatment. Water-in-crude oil emulsion destabilization basically involves three steps, namely flocculation, followed by sedimentation of water droplets due to density differences, and finally coalescence of the individual water droplets. Large droplet sizes, high density difference between the oil and the aqueous phase, and low continuous phase (oil) viscosity causes high sedimentation rates.

Flocculation is the aggregation of two or several droplets, touching only at certain points, and with virtually no change in total surface area. Coalescence is the process of droplets fusing together and forming larger and larger droplets, until the oil and water is separated into two discrete phases.

1.2 Problem Statement

Nowadays, the formation of crude oil and water emulsion has become a challenger problem to the engineer. As we already know, emulsions are really difficult to treat and cause a number of operational problems such as tripping of separation of off-spec crude oil and creating high pressure drops in flow lines. It is essential to treat the crude oil first before transporting and refining them.

Not only that, we must think of some methods that can give us the lowest cost in separating the crude oil and water. As the oil price increase drastically, it is convenient to reduce the processing cost to separate oil and water emulsions. And of coz, the most simplest and efficient method can also reduce the time in separating the emulsions.

On the other perception, if we can minimize the water levels in the crude oils it can reduce the corrosion in the pipeline and also will maximize the pipeline usage. So, in order to solve these problems, we must find the most efficient way in stabilizing the emulsion with the best method available.

1.3 Objectives

The purpose of this study is to extend the study of emulsions to systems containing inorganic solids and crude oil or crude-oil like phases. This work will provide useful information regarding the role of the inorganic solid particle in resolution of emulsion problems facing nowadays in the petroleum industry. This study also investigated the role of asphaltene on water-in-crude oil emulsion stability. The main issue that to be addressed are the influence of asphaltene aggregation, as controlled by resin concentration and solvent quality, and the effect of inorganic solid particles size, wettability, and surface chemistry on the stabilization effectiveness of the inorganic solid particles in water-in-crude oil emulsion systems. And lastly to develop a better understanding of emulsion stabilization in the presence of inorganic solid particles based on different types of inorganic solid particles use and different types of crude oil that will be tested.

1.4 Scope of Study

The scopes of this study are;

1. To study the effect of inorganic solid particles based on percent of water resolved in water-in-crude oil emulsion stability.
2. To characterize inorganic solid particle based on their ability to stabilize water-in-crude oil emulsion.

3. To choose the most stable crude oil, base on their stability after tested with different kind of inorganic solid particle.

1.5 Rationale and significance

Due to the emulsion problem that usually corrode refinery equipment, there is need to find what factor that can affect the stability of water and crude oil emulsion. The main objective of this study is to find the most effective inorganic solid particles that can stabilize water in crude oil emulsion. Other than that, the study also investigates the most stable crude oil by comparing their asphaltene content. And by this, it is hoped that the results will provide an useful information to aid in the production and refining operations of the petroleum industry.

CHAPTER 2

LITERATURE REVIEW

2.1 Crude oil composition

Crude oil is a complex mixture of hydrocarbons, with small amounts of sulphur, oxygen and nitrogen, as well as various metallic constituents, particularly vanadium, nickel, iron and copper. To determine the exact structure and composition of the various components is thus a daunting task, and the selection of fractionation procedure depends on the information desired.

The asphaltene content of petroleum is an important aspect of fluid processability. The SARA method, where the asphaltenes are separated as a group, is therefore often used to conveniently separate the crude oil into four major fractions: saturates (including waxes), aromatics, resins and asphaltenes (SARA), based on their solubility and polarity as shown in Figure 2.1.

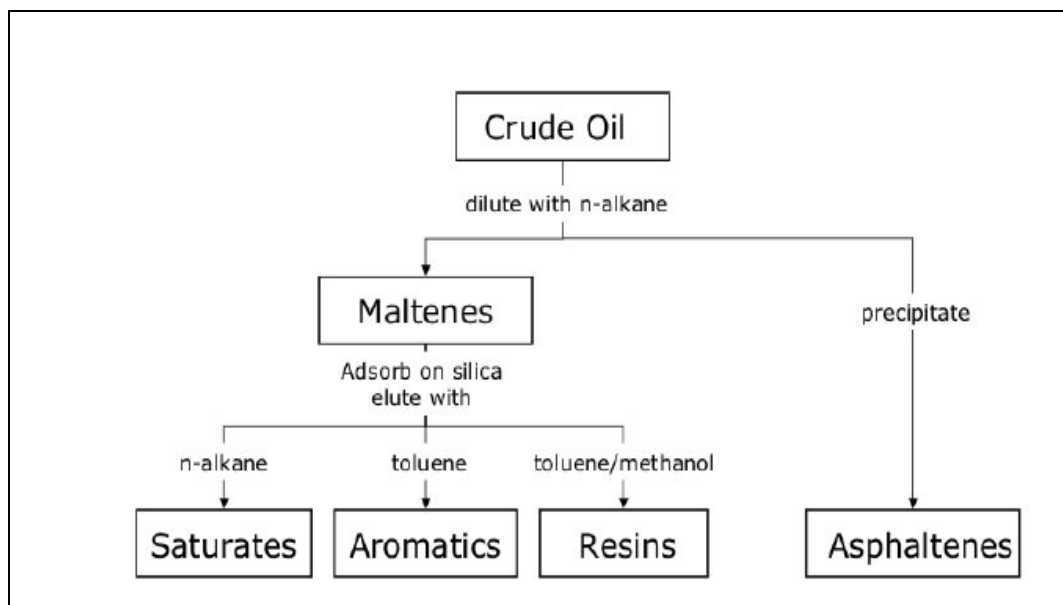


Figure 2.1 Typical scheme for separating crude oil into saturate, aromatic, resin and asphaltene (SARA) components.

The basis for the method is that asphaltenes are removed by precipitation in a paraffinic solvent, and the deasphalted oil is separated into saturates aromatics and resins by chromatographic fractionation. Of the four classes of compounds, only the saturates are easily distinguishable from the rest of the hydrocarbons in the mixture. The absence of π -bonds allows them to be readily differentiated from the aromatic components by virtue of the difference in their polarities. The remainder of the oil is composed of aromatics and heteroatomic compounds of varying degree of condensation, alkyl substitution and functionalism, which constitute a compositional continuum with respect to molecular weight and polarity. The many variations in the recommended procedures may all have some influence upon yield and chemical nature of the fractions. The properties of asphaltenes, for example, have shown to be affected by temperature, precipitating solvent, solvent-to-oil ratio and separation time.

2.2 Asphaltene Chemistry

The word asphaltene was coined in France by Boussingault in 1837. Boussingault described the constituents of some bitumens found at that time in eastern France and in Peru. He named the fraction of distillation residue, which was insoluble in alcohol and soluble in essence of turpentine, “asphaltene”, since it resembled the original asphalt. The strong interest in developing a better understanding of the solution behaviour of asphaltenes, has been motivated by their impact on production, transportation, refining and utilization of petroleum. The asphaltene fraction is composed of the heaviest and components in crude oils. Separated solid asphaltenes usually appears brown to black in color and has no definite melting point but decomposes when the temperature exceeds 300-400 °C. It has been shown that changes in temperature, pressure and oil composition can cause asphaltene precipitation.

Asphaltenes are operationally defined as the non-volatile and polar fraction of petroleum that is insoluble in n-alkanes (i.e. pentane or heptane). As a result, asphaltenes constitute a solubility class of crude oil components, rather than a chemical class. The molecular weight, polarity and aromaticity of precipitated asphaltenes generally increase with increasing carbon number of n-alkane precipitant. A schematic diagram representing the range of heavy compounds precipitated by mixing crude oil with n-pentane and n-heptane is shown in Figure 2.2.

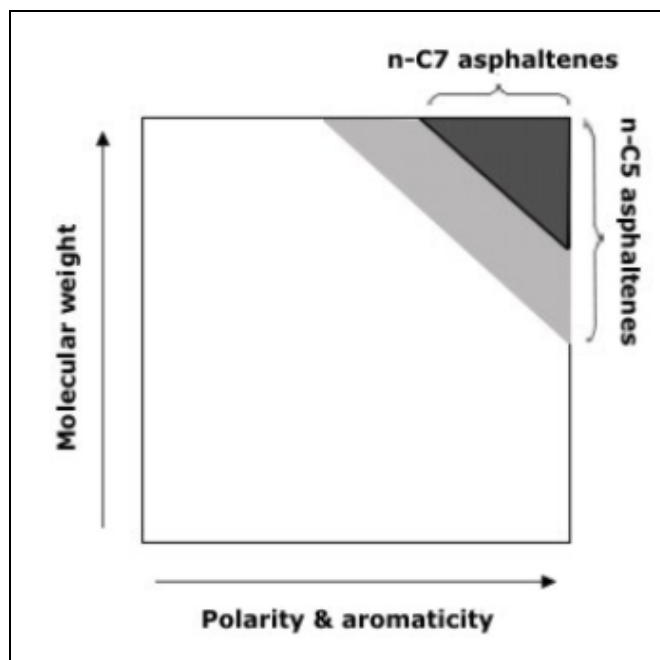


Figure 2.2 Hypothetical diagram representing the molecular characteristics of the asphaltenes precipitated from petroleum by n-alkane addition.

A number of investigators have constructed model structures for asphaltenes, resins, and other heavy fractions based on physical and chemical methods. Physical methods include IR, NMR, ESR, mass spectrometry, X-ray, ultra-centrifugation, electron microscopy, small angle neutron scattering, small angle X-ray scattering, quasi-elastic light scattering spectroscopy, VPO, GPC, etc. Chemical methods involve oxidation, hydrogenation, etc.

While asphaltenes are recognised to be remarkably polydisperse in heteroatomic functionality, molecular weight, and carbon backbone structure, some common features have been established. Asphaltenes are characterized by fused ring aromaticity, small aliphatic side chains, and other elements including sulphur, oxygen, nitrogen, and metals such as vanadium and nickel. The heteroatoms accounts for a variety of polar groups such as aldehyde, carbonyl, carboxylic acid and amide, which are found in the asphaltene molecules. The aromatic carbon content of asphaltenes is typically in the range of 40 to 60 %, with a corresponding H/C atomic ratio of 1.0-1.2. A large percentage of these aromatic carbon rings are

interconnected in the molecular structure and, consequently, the asphaltene molecule appears flat or planar. Figure 2.3 shows a suggested asphaltene structure.

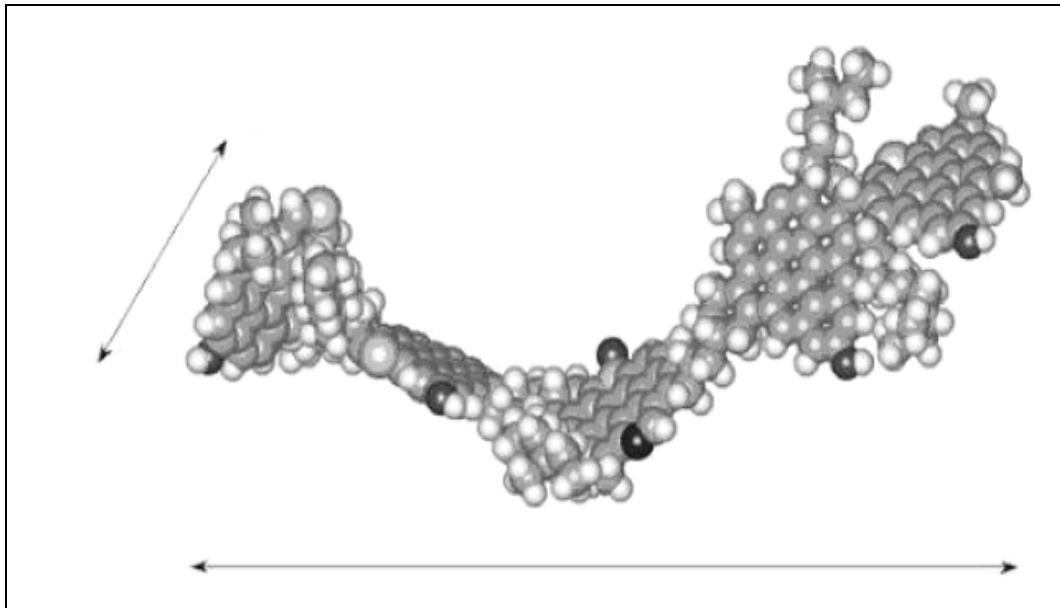


Figure 2.3 Hypothetical molecular structures of the asphaltenes. By courtesy of the Statoil DART (Downhole Asphaltene Remediation Technology) program.

Yen and co-workers proposed a macrostructure model, where the asphaltenes was depicted as stacks of flat sheets of condensed aromatic systems, which was interconnected by sulphide, ether, or aliphatic chains. Espinat et al. suggested the asphaltene molecules to be disc-like with polyaromatic fused ring cores containing polar functional groups. It is currently accepted that asphaltenes consist of aromatic compounds with π - π interactions, which undergo acid-base interactions and self associate through hydrogen bonding.

Several major problems associated with the recovery and refining of petroleum, are related to the aggregation and precipitation of asphaltenes. Investigations have shown that asphaltene particles may self-associate, and form aggregates in the presence of aromatic hydrocarbons. The degree of association is largely dependent upon the aliphatic/aromatic ratio of the solvent. Due to the

aggregation phenomena, measurements of the true molecular weight and the aggregate size are inherently difficult and have resulted in numerous research efforts. The size of the aggregate structure has been suggested to lie between 2 and 25 nm in diameter. The molecular weight obtained have ranged from a few hundred to several million gmol^{-1} , however, the most recent values from several different types of asphaltenes tend to suggest values from 600-1500 gmol^{-1} .

The aggregation is thought to occur through hydrogen bonding, however there is some inconsistency in the description of asphaltene self-association, and both micelles and colloids are used in reference to asphaltenes. An asphaltene colloid is defined as a submicron particle consisting of several asphaltene molecules bound by π -bond interactions between polyaromatic clusters. Asphaltene micelles are considered analogous to a surfactant micelle, where the association of molecules is driven by hydrophobic-hydrophilic interactions. The terms “aggregate” and “micelle” are often used interchangeably in the literature. It has been shown that resins are essential in dissolving the asphaltenes in the crude oil. They are thought to attach to the asphaltene micelles/aggregates with their polar groups, and stretch their aliphatic groups outward to form a steric-stabilisation layer around asphaltenes. However, there still remains the debate about whether the micelle in petroleum is homogeneous insofar as it is composed only from asphaltenes, or if both asphaltene and resin molecules constitute a mixed micelle.

Resins are defined as the non-volatile and polar fraction of crude oil that is soluble in n-alkanes (i.e., pentane) and aromatic solvents (i.e., toluene) and insoluble in liquid propane. They are structurally similar to asphaltenes, on the other hand, molar mass is lower, hydrogen/carbon ratio higher, and the heteroatom content lower. Long et al. showed that once resins were removed from the crude by adsorption chromatography, the remaining oil phase could no longer stabilise the asphaltenes.

Asphaltenes are also known to self-associate due to pressure depletion. At high pressures in the reservoir, the asphaltenes are dissolved in the monophasic crude oil. When the pressure is reduced the molar volume and the solubility parameter

difference between asphaltenes and the crude oil increases towards a maximum at the bubble point of the crude oil. As a result of the reduced solvating power, the asphaltenes may start to precipitate at some onset pressure higher than the bubble point. Prior to the precipitation a stepwise association of the asphaltene molecules will take place. The final precipitation is due to a strong attraction between the colloidal particles and the formation of agglomerates. Once gas evolves, the light alkane fraction of the liquid phase is reduced, and thereby the solvating power for asphaltene molecules increases. The relative change in asphaltene solubility has been shown to be highest for light crude oils that are undersaturated with gas, and which usually contain only a small amount of asphaltenes. This gives the surprising result that light reservoir oils, which are low in asphaltenes are considered to be more likely to experience asphaltene related field problems than heavier, less undersaturated, asphaltenic oils.

A possible way of avoiding asphaltene precipitation is by adding chemicals that act in a way similar to resins by dispersing the asphaltenes in solution. Gonzales et al. investigated the peptization of asphaltenes in aliphatic solvent by various oil-soluble amphiphiles including long-chain alkylbenzene, alkyl alcohol, alkylamine and p-alkylphenol. They found that the head group of the amphiphile influenced the effectiveness of the amphiphiles. Chang and Fogler, using a series of alkylbenzene-derived amphiphiles as the asphaltene stabilisers, investigated the influence of the chemical structure on the asphaltene solubilisation and the strength of the amphiphile-asphaltene interactions. The results showed that the polarity of the amphiphile head group and the length of the alkyl tail controlled the amphiphile effectiveness. Increasing the acidity of the amphiphile head group could promote the amphiphile ability to stabilise asphaltenes, probably through acid – base interactions between the asphaltene and the amphiphiles. León et al. showed results from adsorption studies on asphaltene particles, where the adsorption isotherms of two amphiphiles (nonylphenol and nonylphenolic resin) were compared to a native resin. The adsorption isotherm for the natural resins was characterised by the continuous increase in the amount of adsorbed resins, and there was no indication of a plateau similar to the ones shown by the amphiphiles. This type of isotherm was explained

by the penetration of substrate micropores by resin molecules, which lead to the partial breakdown of the asphaltene macrostructure.

In addition to the resins, other molecules in the petroleum mixture have also shown a tendency to stabilise the asphaltene particles/aggregates. Auflem et al. showed that natural and synthetic naphthenic acids have a tendency to disperse the asphaltenes, and reduce the asphaltene particle size. This was proposed to occur through acid-base interactions between the naphthenic acids and asphaltenes, whereby the naphthenic acid would disperse the asphaltenes in solution in a similar way as the resins.

Naphthenic acids are classified as monobasic carboxylic acids of the general formula RCOOH , where R represents a cycloaliphatic structure. The classification contains a wide variety of structures with carbon number from C10 to C50, and from 0 to 6 saturated rings. In crude oil production, the problems related to naphthenic acids arise from the processing conditions. As the pressure drops during production and carbon dioxide is lost from solution, the pH of the brine increases, which in turn leads to dissociation of the naphthenic acid ($\text{RCOOH} \rightarrow \text{RCOO}^- + \text{H}^+$). As a result, the following may occur: i) deposition of naphthenates in oil/water separators, desalters, tubing or pipelines following complexation of naphthenic acids with metal cations present in the aqueous phase and, ii) formation of stabilised emulsions due to naphthenic acids and naphthenates accumulating at the w/o interface and thereby stabilising colloidal structures.

2.3 Emulsions and Emulsion Stability

Emulsions have long been of great practical interest due to their widespread occurrence in everyday life. They may be found in important areas such as food, cosmetics, pulp & paper, pharmaceutical and agricultural industry. Emulsions are also found in the petroleum industry, where they are typically undesirable and can result in high pumping costs, reduced throughput and special handling equipment.

An emulsion is usually defined as a system consisting of a liquid dispersed in another immiscible liquid, as droplets of colloidal sizes ($\sim 0.1\text{-}10\ \mu\text{m}$) or larger. If the oil is the dispersed phase, the emulsion is termed oil-in-water (o/w) emulsion; conversely, if the aqueous medium is the dispersed phase, it is termed water-in-oil (w/o) emulsion. This classification is not always appropriate and other types as, for instance, multiple emulsions of the type o/w/o, may also be found. In the emulsified state, the interfacial area between the dispersed droplets and the bulk phase represents an increase in the systems free energy. Consequently, the emulsions are not thermodynamically stable, and will seek to minimise the surface area by separating into the different phases. For an emulsion to separate, the droplets must merge with each other, or with the homophase continuum that gradually forms.

Processes that facilitate the separation are sedimentation/creaming, flocculation and coalescence, as shown in Figure 2.4.

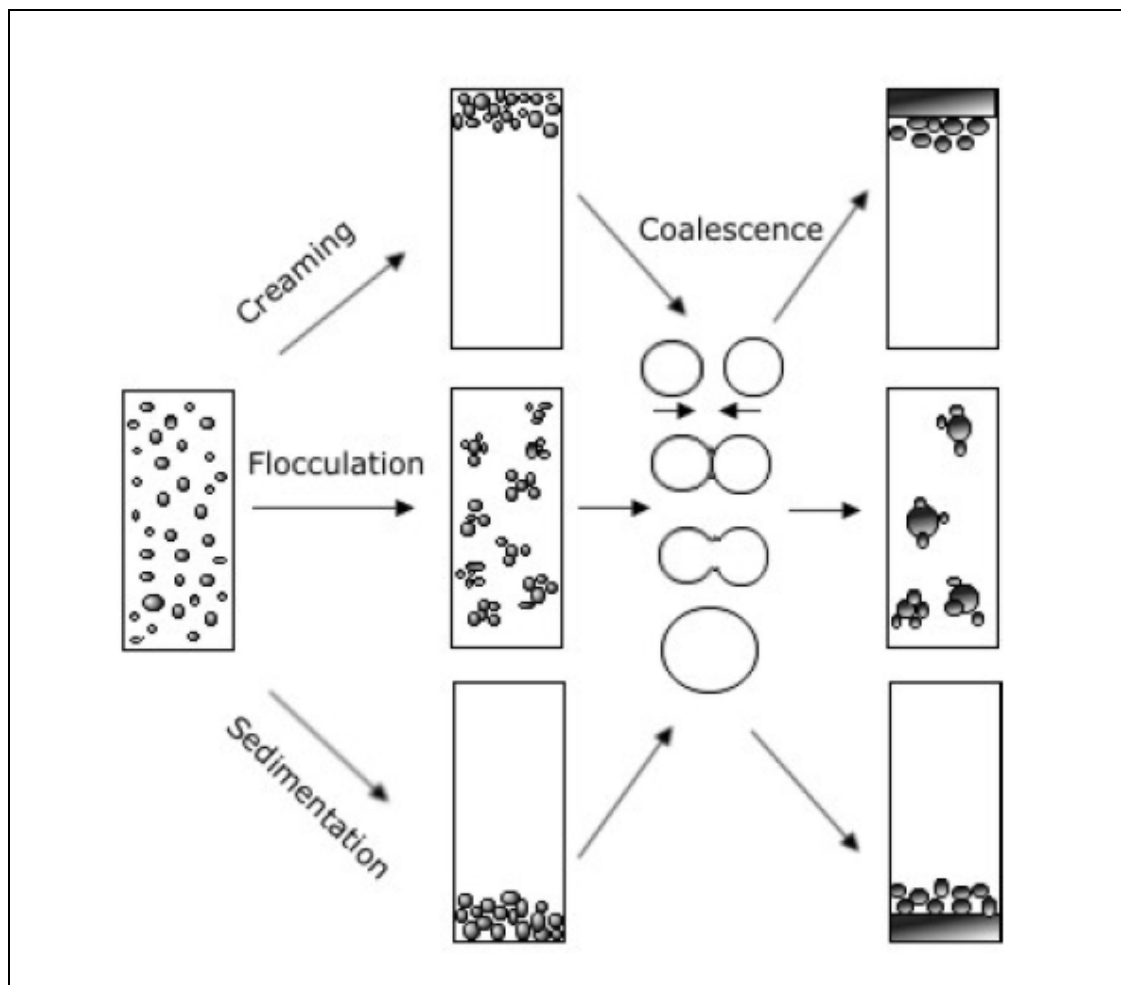


Figure 2.4 Processes taking place in an emulsion leading to emulsion breakdown and separation.

Creaming and sedimentation create a droplet concentration gradient due to a density difference between the two liquid phases, which result in a close packing of the droplets. Aggregation of droplets may be said to occur when they stay very close to one another for a far longer time than if there were no attractive forces acting between them. The size and shape of the individual droplets are for the most part retained. The mechanism of coalescence occurs in two stages; film drainage and film rupture. In order to have film drainage there must be a flow of fluid in the film, and a pressure gradient present. However, when the interfacial film between the droplets has thinned to below some critical thickness, it ruptures, and the capillary pressure difference causes the droplets to rapidly fuse into one droplet. Hence, the properties of the thin film are of uttermost importance for the separation. If the droplets deform, the area of the interface increases and consequently the drainage path in the film also increases, resulting in lower drainage rates.

Electrical double layer repulsion, or steric stabilization by polymers and surfactants with protruding molecular chains, may prevent the droplets to come into contact with each other. Also, polymers, surfactants or adsorbed particles can create a mechanically strong and elastic interfacial film that act as a barrier against aggregation and coalescence. A film of closed packed particles has considerable mechanical strength, and the most stable emulsions occur when the contact angle is close to 90° , so that the particles will collect at the interface. Particles, which are oil-wet, tend to stabilize w/o emulsions while those that are water-wet tend to stabilize o/w emulsions. In order to stabilize the emulsions the particles should be least one order of magnitude smaller in size than the emulsion droplets and in sufficiently high concentration.

Other factors that usually favor emulsion stability are low interfacial tension, high viscosity of the bulk phase and relatively small volumes of dispersed phase. A narrow droplet distribution of droplets with small sizes is also advantageous, since polydisperse dispersions will result in a growth of large droplets on the expense of