AMERICAN UNIVERSITY OF BEIRUT

ADSORPTION OF SURFACTANTS IN THE PRESENCE OF TWO FLUIDS FOR ENHANCED OIL RECOVERY: EXPERIMENTAL INVESTIGATION USING THE QCM-D

by
NOHA EL SETT

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AN ABSTRACT OF THE THESIS OF

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Title: Adsorption of surfactants in the presence of two fluids for Enhanced Oil Recovery: Experimental investigation using the QCM-D

Rock wettability when in contact with two fluid phases - oil and water - plays an important role in oil recovery from hydrocarbon reservoirs. Water injection as a pressure maintenance mechanism performs substantially better in water wet reservoirs compared to oil reservoirs. This is due to low relative permeability to water and tendency of water to imbibe and displace in a water-wet system. A whole category of Enhanced Oil Recovery (EOR) processes consists in altering the rock wettability towards more water wetting used in the Chemical EOR industry. Surfactant water injection is one of these processes.

In this project, the adsorption of surfactants on silica surfaces saturated with oil is examined using the Quartz Crystal Microbalance with dissipation (QCM-D) technique. This mineral mimics sandstone reservoirs. The effect of Sodium Dodecyl Sulfate (SDS) and Triton X-100 concentration is experimentally investigated, along with the effect of salinity. One light crude oil (crude oil A) and one moderately heavy crude oil (crude oil B) were used for the desorption process.

The maximum amount of oil desorbed from the silica surface has reached 27.4% and 22.6% using SDS and Triton X-100 respectively. These values are around the CMC. In addition, SDS did not help in reaching higher amount of desorption when adding salt to the medium. However, using Triton X-100, the total percentage of oil desorption has increased from 87.3% to 92.9% upon addition of NaCl.

As the concentration of surfactant increases more crude oil B has been desorbed from the silica surface until reaching the CMC value where no more desorption occurs. In addition, increasing the salinity of the control solution has decreased the CMC of two surfactants leading to reaching the maximum desorption using lower surfactant concentration. On another note, SDS, an anionic surfactant has led to more desorption of oil from a negatively charged surface than the nonionic surfactant Triton X-100.

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NOMENCLATURE

EOR	Enhanced Oil Recovery
QCM-D	Quartz Crystal Microbalance with Dissipation
SDS	Sodium Dodecyl Sulfate
CMC	Critical Micelle Concentration
DMF	Dimethylformamide
FTIR	Fourier-Transform Infrared spectroscopy
OIIP	Oil initially in place
CTAB	Cetyltrimethylammonium bromide
IFT	Interfacial Tension
N	Newton
M	Molar
Atm	Atmosphere
MHz	MegaHertz
F	Frequency
ODE	Oil Desorption Efficiency
CPP	Critical Packing Parameters
API	American Petroleum Institute
cP	centiPoise
SFT	Surface Tension
CA	Contact Angle
d	Dissipative
p	Polar
OWRK	Owens, Wendt, Rabel and Kaelble
wt%	Weight percent
V	Volume
DW	Distilled water
LS	Low salinity
MS	Moderate salinity
HS	High salinity
UV	Ultraviolet
h	Hour
rpm	Revolutions per minute
s	Second
µS/cm	MicroSiemens per centimeters
TX	Triton X-100
W	Weight
Hz	Hertz
D	Dissipation

CHAPTER I

THEORETICAL BACKGROUND

A. Enhanced Oil Recovery:

According to Xu et al, around 33% of the original oil in place (OIIP) is recovered using primary and/or secondary recovery procedures, hence leaving approximately 60-70% as reserves [1]. Primary recovery, the first step in extracting oil from wells without the addition of any substances, help in recovering only 14% of the oil [2]. Then secondary oil recovery is performed where water is introduced to recover another 18% of the oil. Since these processes could not yield more than one-third of OIIP, tertiary recovery, also called Enhanced Oil Recovery (EOR), is needed to recover more oil. EOR processes drift the oil to the production wells and consequently increase the production rate in the field [2]. Three different types of EOR are present: chemical injection, steam injection (thermal EOR) and miscible gas injection.

The main objective of EOR is to change the mobility of the oil left in the reservoir after primary and secondary recovery. The oil is therefore trapped in the pores of the reservoir due to viscous and capillary forces [3]. Chemical injection is the injection of a certain liquid chemical that can lead to a change in the characteristics of the phase behavior to help in displacing the oil. The addition of surfactants is one of the main chemical injection processes. Alkaline flooding is another process where alkaline will react with different oil components to create surfactants *in situ*. (figure 1).

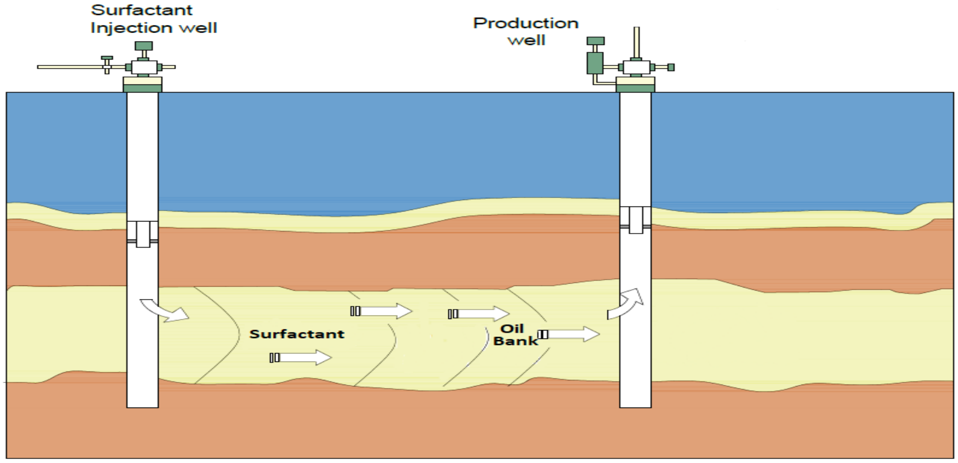


Figure 1: Chemical Enhanced Oil Recovery

At the reservoir conditions, the injection of a certain gas that can be miscible with the oil in order to displace it, is called "Miscible gas injection" and is shown in figure 2. One of the main examples is the injection of miscible CO_2.

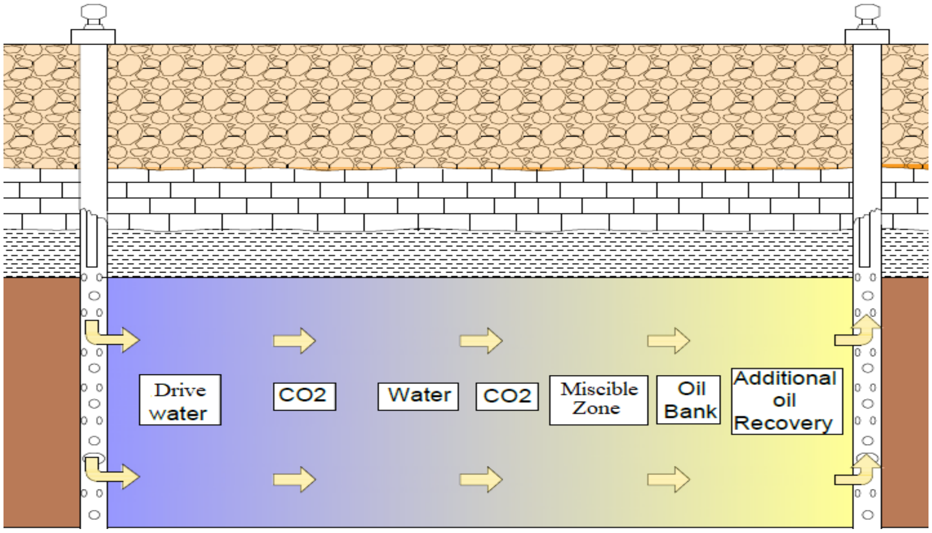


Figure 2: Miscible gas injection

Thermal EOR, presented in figure 3, is the injection of a hot phase such as steam to reduce the viscosity of oil. This process is usually applied for heavy oil where the increase in temperature will lead to a decrease in the viscosity of the trapped oil and therefore the flow to the production wells will become easier.

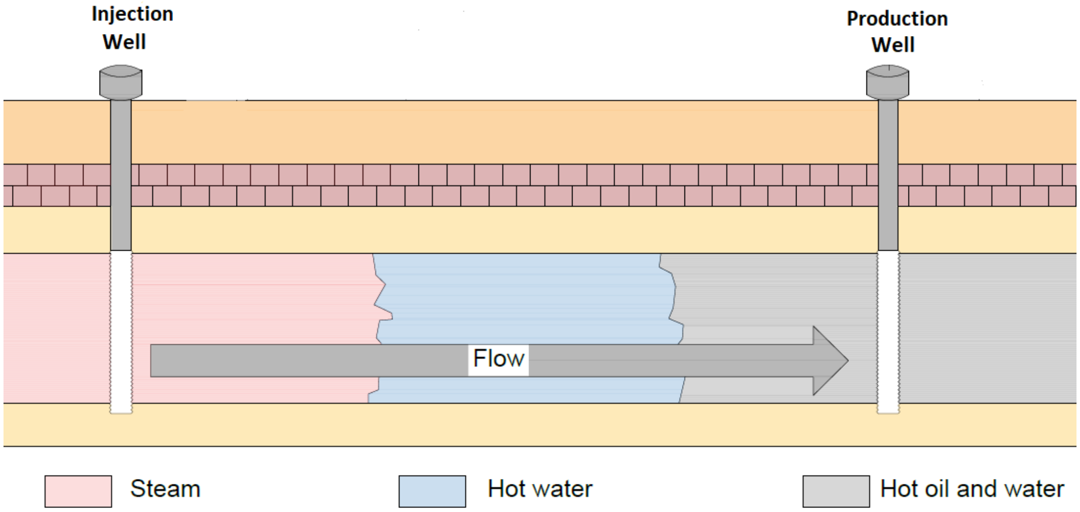


Figure 3: Thermal Enhanced Oil Recovery

B. Surfactant

Surfactants are surface-active substances that can adhere to a surface or an interface and alter its properties. The adsorption of surfactants on mineral surfaces has a crucial role in different fields such as lubrication, oil recovery, process of waste, waterproofing, etc.[4]. By adhering and aggregating at the solid interface, these surfactants alter the interfacial tension between fluids. The application of surfactants is based on their molecular characteristics, i.e. their hydrophobic tail, which is the hydrocarbon (nonpolar) section formed of 6-22 carbon atoms, and the hydrophilic head, which is the ionic (polar) section

[5]. Mainly, the properties related to surface-active components is given by the stability between the hydrophobic and hydrophilic sections of a surfactant [3]. The surfactant structure is presented in figure 4.

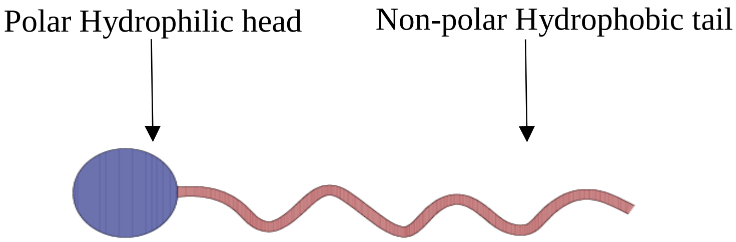


Figure 4: Surfactant structure

1. *Classification of surfactants:*

The nature of the hydrophilic group will categorize the synthetic surfactants accordingly. They can be either anionic, cationic, nonionic or zwitterionic:

a. The anionic surfactant has a sulfate group, a carboxylic group or a sulfonate group for the hydrophilic section [6]. It dissociates in aqueous solution and lead to a negative charge hence called anionic. The anionic type is an operative agent for sandstone reservoirs [7]. They can lower the interfacial tension to very low standards where the capillary pressure is almost zero [8]. They display low adsorption on the rock of the reservoir, and they can be economically synthesized which make them the most used in industry. Examples: Sodium dodecyl sulfate, sodium dodecyl benzene sulfonate.

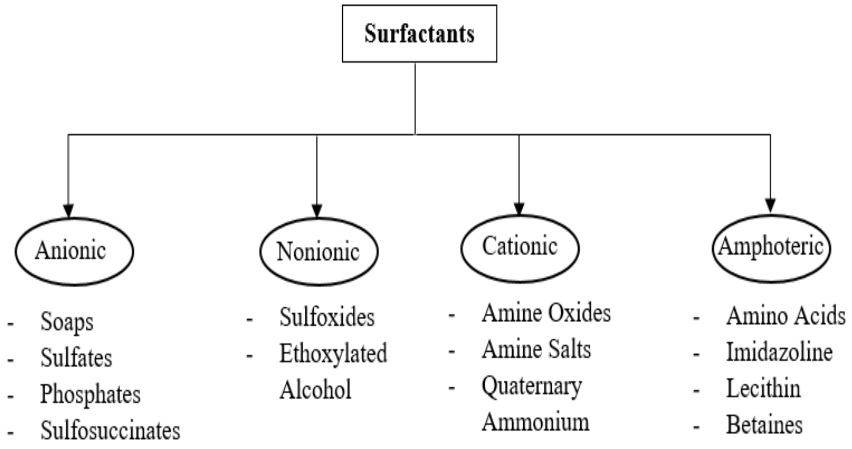
b. The cationic surfactant has a quaternary ammonium group for the hydrophilic head [6]. It dissociates in aqueous solution and lead to a positive charge hence called cationic. It forms stable solutions and it is the steadiest candidate in carbonate reservoirs [7]. Examples: Cetyltrimethylammonium bromide (CTAB), dodecylamine hydrochloride.

c. The nonionic surfactant has a polypeptide, a polyoxyethylene or a sucrose hydrophilic head [6]. It does not ionize in aqueous solution and the hydrophilic part is greater than the hydrophobic part. The nonionic molecule is effective in hard water or brine with high salinity [7]. They are also used as co-surfactants because they have a low ability to decrease the interfacial tension [2]. Example: Polyethylene oxides.

d. The zwitterionic surfactant has both the anionic and cationic portions attached to the same particle. There is no noteworthy research about this type of surfactants in enhanced oil recovery [7]. However, Zhang et al. (2015) proved that this type of surfactants has a strong tolerance to electrolyte, resistant to temperature and it has better wetting performance [7].

These four main categories are presented in table 1.

Table 1: Classification of surfactants



The adhesion of surfactants on the solid surface is mainly determined by the electrostatic interaction between the charged head group of the surfactant and the inherent charge of the surface when present in an aqueous phase [4].

2. *Application of surfactants*

Surfactants are applied in a variety of applications from the purification of raw substances to the quality improvement of cosmetics, dyes and pharmaceuticals. Surfactants can be used in agrochemicals, processing foods, paints, mineral ores, lubricants, laundry products, etc. They can also play a role in medical and biological applications, as well as in health and safety [9]. Some of the important sections of the surfactant application are presented in figure (6). According to the different applications where surfactants are being used, the wanted properties such as solubility, critical micelle concentration, wetting control may considerably differ [10]. In other words, specific characteristics are needed for different applications. Even though the science of surfactants is considered a practically mature discipline, there is always new molecules that need to be designed in order to fulfill some specific applications. One of the first and essential reasons surfactants are being used is their ability to alter the properties of interfaces and surfaces [9]. This property makes them subtle to progress where new products need to be developed to encounter industrial changes for new applications.

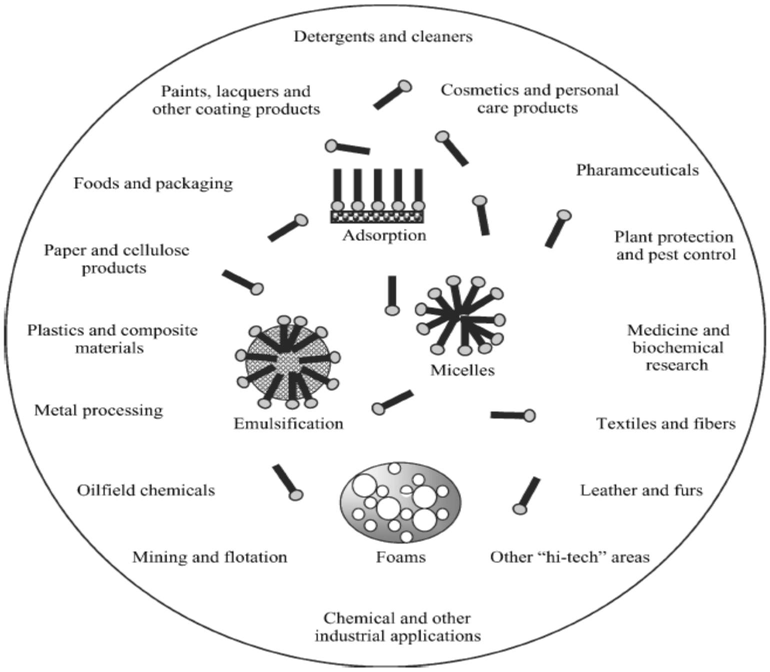


Figure 5: Major applications of surfactant [10]

3. *Adsorption mechanism of surfactants on Solid-Liquid interface*

The performance of surfactants on an interface is mainly determined by the amount of surfactants needed to make a change on the surface and the maximum change it can yield irrespective of the quantity used. The mechanism of surfactant adsorption are mainly determined by three factors: (1) the structure of the adsorbate – surfactant – i.e. whether it is charged or not, whether its tail is long or short, branched or straight; (2) the structure of the solid surface i.e. whether it holds charged sites or it is nonpolar, the nature of the atoms present in these sites; (3) the environment of the solution such as pH, temperature, presence of electrolyte [11].

Surfactants may adsorb on the solid surface according to different mechanisms [11]:

a- Ion exchange where the charged ions of the surfactant will replace the counterions present on the surface from the solution.

b- Ion pairing where the ions of surfactants will adsorb on the oppositely charged locations where counterions are not present.

c- Acid-base interaction via hydrogen bond formation.

d- Hydrophobic attachment where the tendency of the hydrophobic group of the surfactants is large enough to allow them to adsorb on the surface of the solid.

e- Polarization of π electrons where the electron-rich aromatic nuclei of the surfactant, if present, will adsorb on the strongly positive surface of the solid.

The direction in which the surfactant adsorbed on the surface will determine whether the surface will be hydrophobic or hydrophilic: if the surfactant is adsorbed by its hydrophobic group, the surface becomes more hydrophilic, and if the surfactant is adsorbed by its hydrophilic group, the surface becomes more hydrophobic [11].

The change in pH may affect the adsorption of surfactants: when the pH of the solution is lowered, protons will be adsorbed on the solid surfaces making it more positive. Hence, the adsorption of anionic surfactants will be favored. This change in pH can also affect the surfactant containing an ionic group and transform it to a neutral molecule that will adsorb on the surface through hydrogen bonding or dispersion forces [11].

Generally, an increase in the temperature will lead to a decrease in the efficiency of the ionic surfactants being adsorbed on the surface. However, the effect of the temperature is less pronounced than that of the pH [11].

C. Surfactants in Oil Recovery

One of the applications of surfactants is its use in Enhanced Oil Recovery. In chemical injection, the use of surfactants has always been challenging and for decades, extensive efforts were made to design and optimize an appropriate surfactant to reach an effective chemical EOR. The concept of adding surfactants into the reservoir goes back to Uren and Fahmy in the early 1900s [7]. Using surfactants looks like a promising approach to resolve some desires in the oil industry. In EOR, the polar section of the surfactant will interact with water molecules and the nonpolar section will interact with the residual oil leading to oil-in-water or water-in-oil emulsions [12]. When being introduced to the reservoir, the surfactants will be in contact with the oil imprisoned in the pores of the reservoir rock, therefore decreasing the interfacial tension and mobilizing the oil [2]. Consequently, the wettability of the reservoir will be driven to a more water-wet system thus reducing the residual oil saturation and increasing oil recovery [7]. The formation of the microemulsion is due to the injection of primary surfactants. However, in some cases a co-surfactant is added that will improve the efficiency of the primary surfactant either by changing the viscosity of the liquids or by altering the energy of the surface. After the injection of surfactants, a certain polymer can be introduced to regulate the mobility and steady the flow pattern as well as improve the sweep efficiency [12]. For this mixture to be moved smoothly into the producing well, water should be pumped as presented in figure 6 [12].

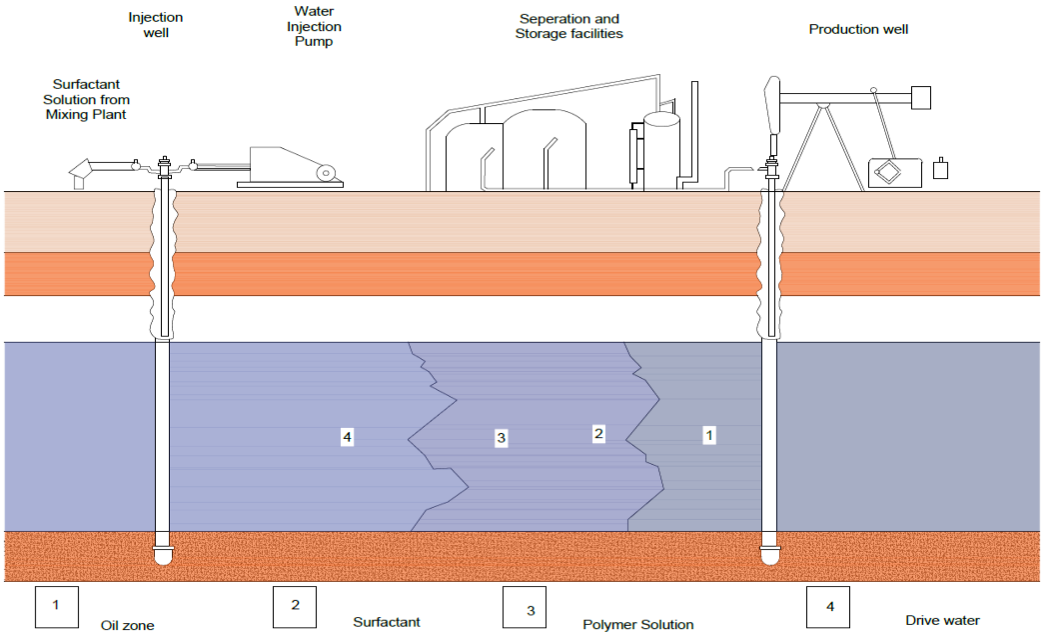


Figure 6: Mechanism of surfactant flooding for the removal of oil [12]

Between oil and water, the interfacial tension will be reduced from 30 mN/m to very low values of 0.01 mN/m or less [13]. Reservoirs have different properties, hence the surfactants used should be well suited to the reservoir conditions such as temperature, pressure and rock minerals in order to reach low IFT. Nonetheless, one of the issues restraining the efficiency is the unwanted loss of surfactants due to their adsorption onto the rock of the reservoir [14]. Different factors can affect the adsorption of surfactant such as their concentration, their type, the characteristic of the rock, and the properties of the bulk solution [13].

Wu et al. (2005) investigated a set of branched alcohol propoxylate sulfate, which is one type of anionic surfactants. Their results show that at low concentration, these surfactants can reach an IFT as low as 0.01 mN/m or less between the brine and the oil. They also showed that the IFT and the adsorption can be affected by the number of propoxylate groups [15].

In EOR, anionic surfactants are the most widely used due to their properties, their stability, their low adsorption on the surface of the reservoir and their economical manufacturing [3]. Nonionic surfactants, which can withstand high-salinity brine, are mainly used as co-surfactants to enhance the performance of surfactants. On the other hand, cationic surfactants are considered a bad option due to their strong adsorption on the rock of the reservoir [3]

1. *Mechanism of oil removal*

Using surfactants in enhanced oil recovery helps in changing the wettability of the reservoir from oil-wet to water-wet system. The more the solid surface becomes water-wet, the better it is for the desorption process. In this case, the oil droplets will have smaller contact angle with the surface and hence the efficiency of the oil displacement will significantly improve [16]. Active substance of oil will adsorb on the solid surface hence, turning it to an oil-wet system. Once they adsorbed on this oil-wet surface, they start spreading on it as it is shown in figure 7.

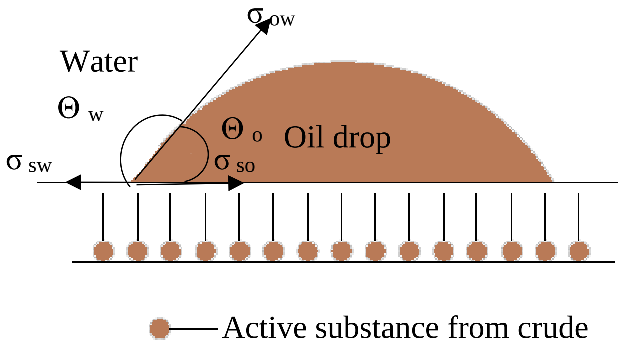


Figure 7: Oil wet surface

In the presence of two fluids – oil and brine – surfactants may adsorb on the solid surface according to different mechanisms. One of the suggested processes is the “roll-up” mechanism which is shown schematically in figure 8.

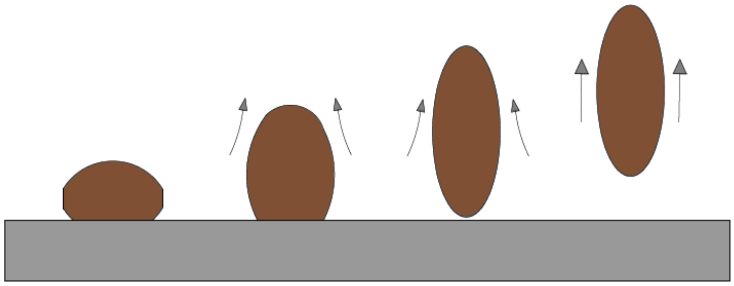


Figure 8: Roll-up mechanism for oil removal

During this process, the contact angle between the oil-water interface and the surface will decrease in order to remove the droplet of oil from the rock [17]. What drives the separation of oil from the surface is the variance of interfacial tensions between the three phases [18]. When the contact angle is greater than 90^{o}, good oil removal is commonly obtained [19] and it will be ideally increased to approximately 180^{o} when “rolling-up” occurs [18].

According to Young’s equation, which is of significance to this type of mechanisms and is presented in figure (10):

$$\sigma_{SO} = \sigma_{SW} + \sigma_{ow} cos\theta_w \qquad (1)$$

Where σ_{SO} is the interfacial tension between the surface and the oil

σ_{SW} is the interfacial tension between the surface and water

σ_{ow} is the interfacial tension between oil and water

θ_w is the contact angle between oil-water interface and the surface

The droplet rolls up when $\sigma_{ow} \cos\theta_w + \sigma_{os} - \sigma_{sw} > 0$ [18].

σ_{ow} and σ_{sw} will decrease due to the adsorption of surfactants on the oil-water interface and on the surface of the film formed by the crude oil respectively. σ_{SO} will remain constant during the process. Therefore, $cos\theta_w$ will increase and hence θ_w will decrease. In this way, the oil droplets will be easily removed and the efficiency will be improved [16].

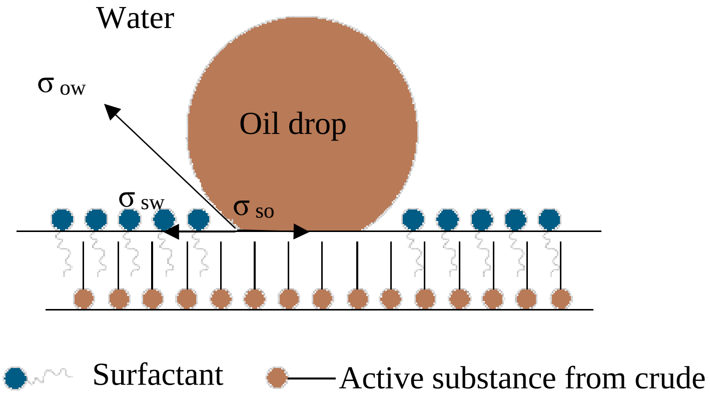


Figure 9: Schematic diagram of the adhesion analysis of the oil droplet

Two supplementary mechanisms are occurring other than the “rolling-up” mechanism: emulsification and solubilization.

Emulsification, which is considered independent of the nature of the rock, comprises an interaction between the oil and the surfactant [19]. The mechanism is shown in figure (11). In this “diffusional” mechanism, the surfactant solution will diffuse between the drop of oil and the rock causing removal of oil [18]. The reduction of the interfacial tension permits the deformation of the oil film in an easy manner and the formation of minor droplets of emulsion [20].

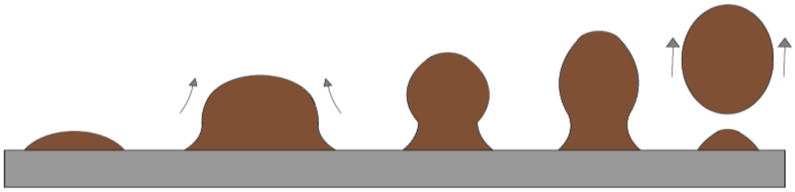


Figure 10: Emulsification mechanism for oil removal

In the solubilization process, which is presented in figure 11, the oil is being soluble into *in situ* made microemulsion independent of the rock surface [19]. Miller and Raney define the solubilization mechanism as being related to the removal of oil with a large amount of polar elements. The interaction between these constituents and the surfactants make liquid crystal grow until the intermediate phase come off into the aqueous phase leaving room for the oil to be in contact with the surfactant solution. In case there is a large amount of surfactant with respect to the oil, direct solubilization into the micelles may occur [20].

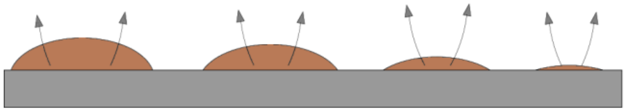


Figure 11: Solubilization mechanism for oil removal

In the last two mechanisms, a chunk of the oil is removed from the surface by making the interface between the water and oil unstable [17].

The change of wettability of oil using surfactants is delicate to different aspects such as the pressure, temperature, size and concentration of surfactants, primary contact angle, charge of particle, charge and roughness of the rock surface, etc. [17].

2. *Formation of Microemulsion*

Microemulsions are a mixture of two immiscible liquid phases – hydrocarbon and water - assisted by a surfactant that can be present alone or with a co-surfactant [10]. This transparent mixture has the potential to decrease the interfacial tension between water and oil to very low values, to lower the viscosity and change the wettability of the system as stated by Zhu et al. (2003) [8]. All of these parameters are important to be able to mobilize the oil. With water or oil, microemulsion can reach very low IFT values in the range of 10^{-3} mN/m and this is the basis of their stability [12]. According to Kayali et al (2010), the performance of oil recovery is best predicted by the performance of the interfacial tension using microemulsion processes [16].

The formation of microemulsion is strongly dependent on the structure and quantity of the surfactants and co-surfactants used. For example, nonionic surfactants can form microemulsions without adding other elements while cationic surfactants need a co-surfactant [16].

At low surfactant concentration, the molecules will start spreading in the form of monomers and as the injections are repeated for EOR, the concentration increases and therefore, aggregation of molecules starts taking place until reaching the critical micelle concentration (CMC). Above this value, formation of micelles occurs [3]. This process is shown in figure 12. In the case where water is the solvent, the formation of micelles will be shaped in a way where the tail section is pointed inward and the head section is pointed outward. If the solvent is hydrocarbon, the surfactant will be oriented in the reversed direction [3].

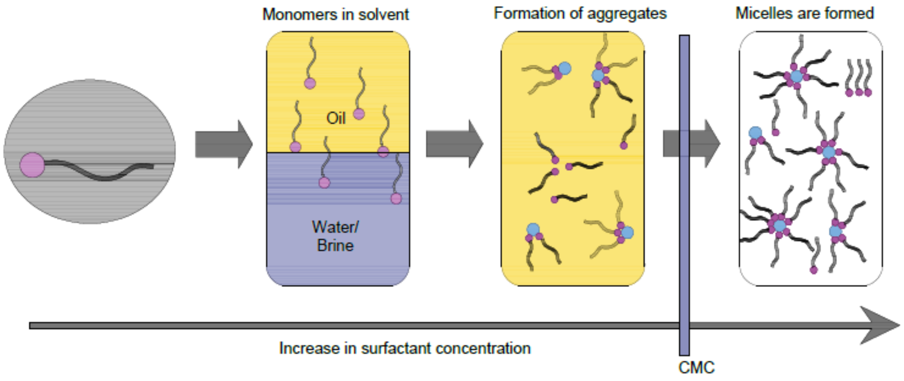


Figure 12: Process for micelle formation

The effect of surfactant adsorption with respect to the surfactant concentration is represented by the Gibbs adsorption isotherm:

$$\Gamma_1 = -\frac{1}{2\,RT}\left(\frac{\partial\gamma}{\partial lnc_1}\right) \qquad (2)$$

Where Γ_1 is the adsorption density of surfactants.

R is the gas constant.

T is the temperature.

$\partial\gamma$ is the surface energy change.

∂lnc_1 is the surfactant concentration change.

The principle of flooding surfactants into the reservoir is to decrease the energy of the surface which is described by equation 2 [12]. This decrease in energy as a function of surfactant concentration for micellization is represented in figure 13.

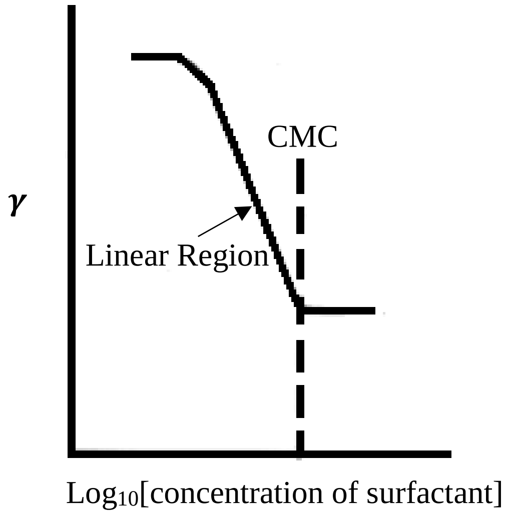


Figure 13: Surface energy as a function of surfactant concentration

Below the CMC value, the surface energy is still decreasing with the increase of surfactant concentrations, which is shown by the linear slope $\partial\gamma/\partial lnc_1$. This constant slope shows that there is no change in the adsorption density when the surfactant concentration is increased. The decreases in surface energy is due to an increase in the chemical potential when continuously adding more surfactants and this is shown in equation 3:

$$\mu_i = \mu_i^o + kTlnc_i \qquad (3)$$

Where μ_i^o is the standard chemical potential of component I (1M for solutes and 1 atm for gas mixtures).

k is Boltzmann constant.

When the pressure, temperature and composition of the elements are considered stable, the elevation of the system's free energy while adding an inconsiderable quantity of an element describe the chemical potential.

Even though the adsorption density remains constant, it will decrease the energy needed to form a new surface, therefore the energy of the surface keeps decreasing until reaching the CMC.

At the CMC, a sudden change happens due to having $\frac{\partial\gamma}{\partial lnc_1} = 0$ (i.e. no adsorption). In this region, micelles start to form and this formation of aggregates takes every supplementary molecules. In this case, the concentration of monomers remains constant [12].

Surfactants have an essential part in forming the precise type of microemulsion that decreases the interfacial tension of oil [21].

The type of surfactant, electrolytes and the rock properties that exist in the mixture determines the adsorption isotherm. Adsorption starts occurring when aggregates start establishing at the surface of the rock and the formation of a monolayer takes place. An extra layer begins to form once the monolayer adsorption reaches equilibrium. When multiple layers start forming, surfactant will be lost remarkably [22].

The loss of surfactant by phase trapping, adsorption on the surface and precipitation was studied a lot by Ahmadall (1993), Somasundaran and Zhang (2006) and then by Lv et al. (2011). This phenomenon reduces the availability to move the trapped oil. Phase trapping is the passage of surfactants in the microemulsion or oil phase due to high salinity and high temperature leading to unmet needs concerning the low IFT conditions [8]. The process of adsorption is due to the structure of surfactant, oil saturation, temperature, salinity and ion exchange. It is one of the main processes to determine the amount of surfactant needed for the EOR process [21].

D. The QCM-D Technique

The Quartz Crystal Microbalance with dissipation (QCM-D) involves a piezoelectric, thin quartz crystal inserted between a pair of electrodes [23]. The principle behind the QCM-D is to keep track of the mass change by checking how the resonance frequency (f) and the dissipation (D) are varying when an AC voltage is being applied. When the quartz sensor is subjected to an electric field, it begins to oscillate at a certain frequency that is associated to the mass of the crystal. This change in frequency is somehow related to mass loading and liquid loading (liquid trapping is being ignored since the crystal surface is smooth) [24]. The mass loading, which is described by the Sauerbrey’s equation is suitable to the conditions that the adsorbed layer is equally distributed, does not slip at the interface, thin and rigid, and goes as follow [23]:

$$\Delta m = -\frac{C\,\Delta f}{n} \qquad (4)$$

Where $C = \frac{t_q \rho_q}{f_0} = 17.7$ ng $Hz^{-1}cm^{-2}$ for a 5 MHz quartz crystal (characteristic constant).

t_q is the thickness of the quartz crystal

ρ_q is the density of the quartz crystal

f_0 is the fundamental frequency of the quartz crystal in air

n = 1, 3, 5, 7 denoting the overtone number.

Δf is the change in frequency.

The QCM-D technique was shown to be highly dependable in order to measure any small change in the nanogram range concerning the adsorption of species on solid surfaces [25]. When adsorption occurs on the surface of the crystal, its mass starts increasing leading to a change in the oscillation frequency. For this reason, researchers are using it to evaluate the chemical EOR methods in a simple and fast way by examining the adsorption/desorption of different components from solid surfaces at the microscopic level [24].

1. Effect of Different Parameters on the Adsorption of Surfactants on Solid Surfaces

Different parameters may affect the adsorption of surfactants:

Surfactant Concentration

A study conducted by Terron-Mejia et.al. showed that at low concentration, association of all surfactants to the hydrocarbon adsorbed on the mineral surface occurs [6]. The surfactant will be present as monomers and there is an equilibrium between these surfactants and the interface [26]. Surfactant micelles start to appear as the concentration is increased while the rest associates with the hydrocarbon molecules. It was realized that there was a competition between self-association and the association with the other molecules. At high concentrations, complete desorption of the hydrocarbon chains occurs since they are enclosed by the hydrophobic tails of surfactants forming a complex with the outer components being the hydrophilic heads of surfactants [25].

Apaydin and Kovscek indicated in their experiments, that very high concentration can lead to deviation from normality: buildup of pressure gradient may occur against the flow direction [2].

Using QCM-D, Ray et al showed in their study that the adsorption of surfactant increases, and hence the frequency decrease, as the concentration of solution increases near the critical micelle concentration (CMC). Past the CMC, the frequency stabilizes [25].

Electrolyte Concentration

Hybrid enhanced oil recovery process was considered using QCM-D. Nourani et al showed that injecting low salinity solutions results in lower surfactant adsorption [14]. As the concentration of electrolyte increases, the adsorption of surfactant reaches a maximum at lower surfactant concentration. In other words, the formation of the monolayer happens at smaller surfactant concentrations [25].

The oil desorption efficiency (ODE), which is the ratio of oil desorbed by surfactant flooding over the oil initially adsorbed, was also calculated from the basis of surfactant adsorption on the surface [14]. After comparing the ODE with and without the low salinity solution, the results showed higher value in the presence of low salinity solution than in its absence [14].

Surfactant Size

By comparing the results using two different surfactants, Nourani et al indicated that the higher the volume tail and the higher the Critical Packing Parameters (CPP) of a surfactant the more the adsorption, in the presence of high electrolyte concentration [14]. The CPP is represented by the following formula:

$$CPP = \frac{V_t}{a_t l_t} \qquad (5)$$

Where V_t is the volume of the hydrocarbon tail, a_t is the head group area and l_t is the hydrocarbon length.

Moreover, the longer the hydrophobic chain, the higher the adsorption [27].

Li et al used the QCM-D technique to show that heavy components are more likely to stay adsorbed on the surface than light components, whatever the type is, due to the strong interaction between oil and the surface [23]. Their results are summarized in figure (14).

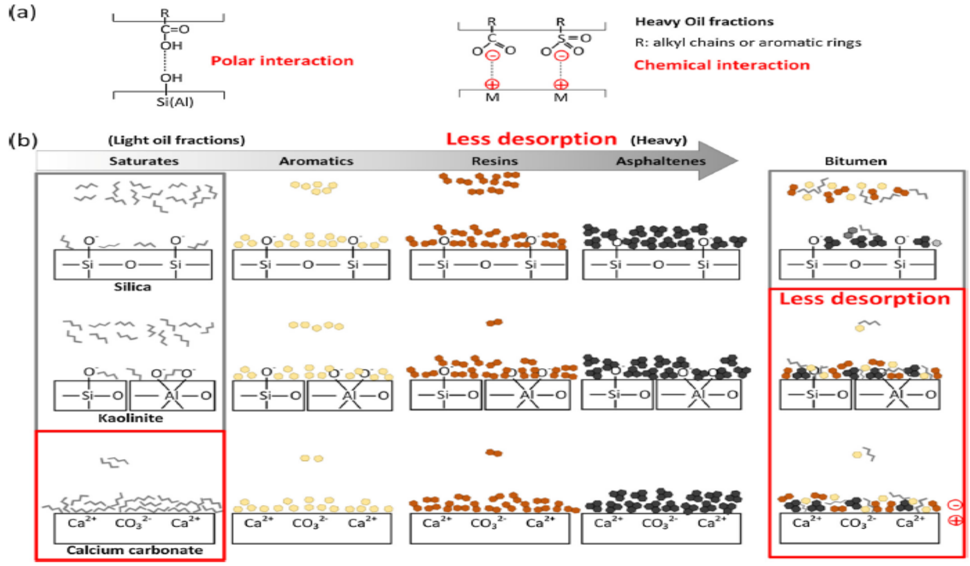


Figure 14: Desorption properties of SARA and bitumen on silica, kaolinite and calcium carbonate in aqueous solutions [23].

Mineral Surface

The mineral composition of the rock has a high impact on the adsorption of surfactant [27]. The mineral surfaces were investigated where the surface with negatively and positively charges show more adsorption than single-charged surfaces [14].

They also showed that the charge of the mineral surface affect the adsorption/desorption process: silica and kaolinite are negatively charged in the contrary to calcium carbonate, this will lead to high interaction between the negatively charged oil fractions and the calcium carbonate surface and therefore the desorption will be much more difficult to occur. Calcium carbonate has the higher affinity toward the oil components, followed by kaolinite and then by silica [23].

Acid Number

Nourani et al also showed that the lowest ODE was for the crude oil with the highest total acid number. Therefore, the desorption increased when acidic components decreased [24].

Inclusion Complex

Romero-Zeron et al evaluated a new concept in preventing the adsorption of surfactant by forming surfactant/β-cyclodextrin complexes. From the dynamic adsorption evaluation, the use of the complex has decreased the surfactant adsorption by 61% [27]. Also the QCM-D measurements show that the adsorption of the inclusion complex is 50% less than the adsorption of the surfactant alone [27].

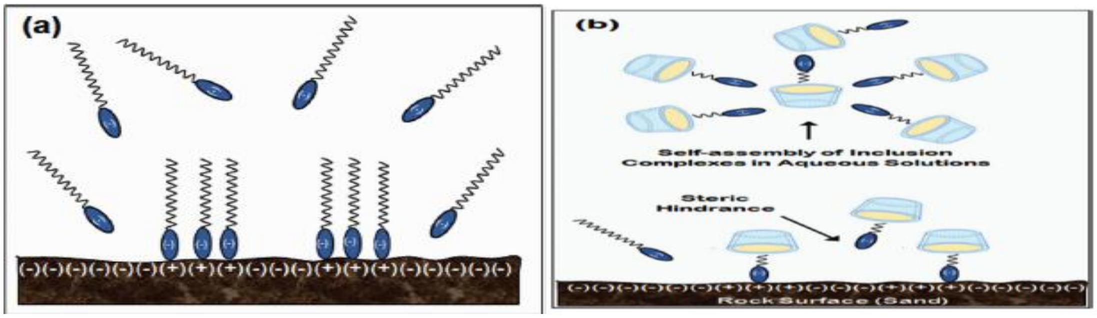


Figure 15: Adsorption of surfactant (a) in free-state and (b) in complex state [2].

CHAPTER II

MATERIAL AND METHODOLOGY

A. Material

1. Chemicals

Two different surfactants were studied in this research: The anionic surfactants sodium dodecyl sulfate (SDS) from Sigma-Aldrich and the nonionic surfactant Triton X-100. Their molecular structure is shown in figures 16 and 17 below and their properties is shown in table 2.

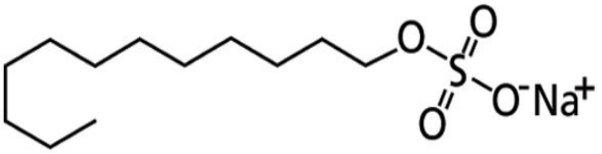


Figure 16: Molecular structure of Sodium Dodecyl Sulfate

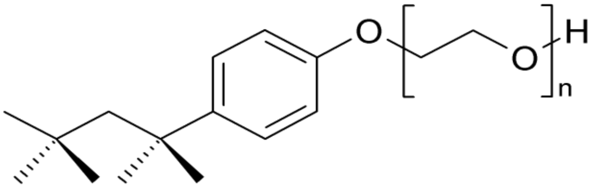


Figure 17: Molecular structure of Triton X-100

Table 2: Properties of the three different surfactants used in the study

	SDS	**Triton X-100**
Chemical formula	$NaC_{12}H_{25}SO_4$	$C_{14}H_{22}O(C_2H_4O)_n$ (n=9-10)
Molar Mass (g/mol)	288.372	647
Density (g/cm^3)	1.01	1.07
CMC (mM) in DW	8.2	0.22-0.24

Three types of oil were used: a synthetic oil which is n-dodecane, $CH_3(CH_2)_{10}CH_3$, with a molecular weight of 170.33 g/mol and a density of 0.75 g/m^3 from Sigma-Aldrich, and 2 types of crude oil imported from Kuwait.

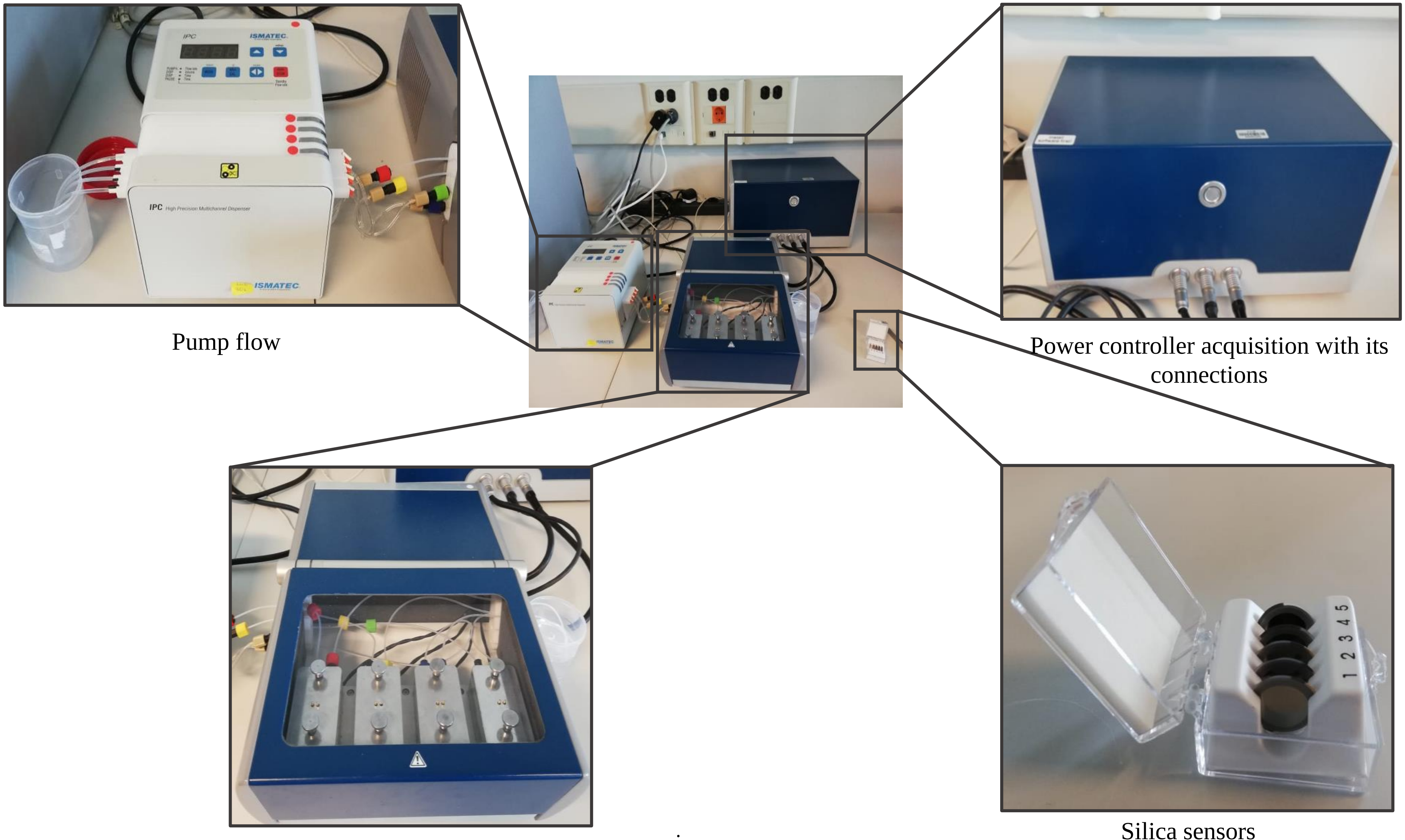
Gold and silica sensors obtained from Qsense Biolin Scientific were needed to mimic the pores of a reservoir. In addition, one kind of mineral nanoparticles, silica, was used to prepare the mineral surface.

Other materials were used in the experiments such as sodium chloride, NaCl, with a molecular weight of 58.44 g/mol to prepare the saline solutions, the N,N-dimethylformamide (DMF) for the surface energy analysis. In addition, ammonia NH_3, hydrogen peroxide H_2O_2, ethanol C_2H_5OH and 2% SDS were used for the cleaning process of the gold and silica sensors.

2. Instruments and equipment

In order to quantify the adsorption of SDS and desorption of oil from the oil-coated sensors and study the microscopic interaction between solids and liquids, the QCM-D instrument from Q-sense was used. This instrument is a technique that enables the measuring of the mass of the particles added or removed in terms of frequency, and the matrix stiffness resulting from the adsorption in terms of dissipation. The different parts of the QCM-D are shown in figure 18.

The instrument will be calibrated with air and the temperature will be kept constant during the experimental run. A control solution such as distilled water or a saline solution will be flushed into the chamber until a baseline is perceived, in this way, the flow rate will remove any material that is not adsorbed to the surface. Different concentration of surfactants will be compared to the control solution in order to determine the amount of oil desorbed from the surface of the sensor.



Pump flow

Power controller acquisition with its connections

4-sensor module

Silica sensors

Figure 18: QCM-D and its components

In order to coat the sensors with the appropriate material in a uniform manner, the spin coating technique was performed. The spin coater used is from Laurell Technologies model number: WS-650MZ-23NPP shown in figure 19.

Figure 19: The spin coater used in the study

3. *Material characterization*

a. <u>Crude oil</u>

The two types of crude oil were imported from a field from Kuwait. Crude oil A, which is lighter than crude oil B, was first filtered using a hydrophobic membrane in order to remove the water present in it.

The densities of crude oil A and B was determined at 21^{o}C with a DMA-35 density meter (RheoSense). The density of the crude oil A was determined to be 0.794 g/ml (API= 46.7) hence making it a light oil, whereas the density of crude oil B was determined to be 0.871 g/mL (API = 30.96) making it a medium oil

In addition, the viscosity was measured at 20°C with a microVISC viscometer (RheoSense) for the crude oil A. The value was determined to be 17.83 cP. For the crude oil B, a FungiLab viscometer was used to obtain a viscosity of 30.224 cP.

Using the OCA 15PRO optical tensiometer, the interfacial tension was determined by the pendant drop technique and it was found to be 24.26 mN/m for crude oil A and 78.51 mN/m for the crude oil B.

The different properties of the two types is summarized in the table below.

Table 3: The different properties of the two oil used in the experiment

	Type A	**Type B**
Density (g/mL)	0.794	0.871
API (⁰)	46.7	30.96
Viscosity (cP)	17.83	30.224
IFT (mN/m)	24.26	78.51

b. Silica sensor

The surface free energy of the silica sensor was determined using the optical tensiometer by getting the surface tension (SFT) of water and N,N-Dimethylformamide (DMF) and their contact angle (CA) on the surface. The dissipative (d) and polar (p) components for these two components was also determined by the optical tensiometer. From the OWRK calculation method, the surface free energy of the silica sensor was 50.07 mN/m. The final results are shown in table 4.

Table 4: The total and component surface tension of water and DMF

	SFT (total) (mN/m)	**SFT (d) (mN/m)**	**SFT (p) (mN/m)**	**CA (^{0})**
Distilled water	72.60	26.00	46.80	50.09
DMF	37.35	32.42	4.88	39.94

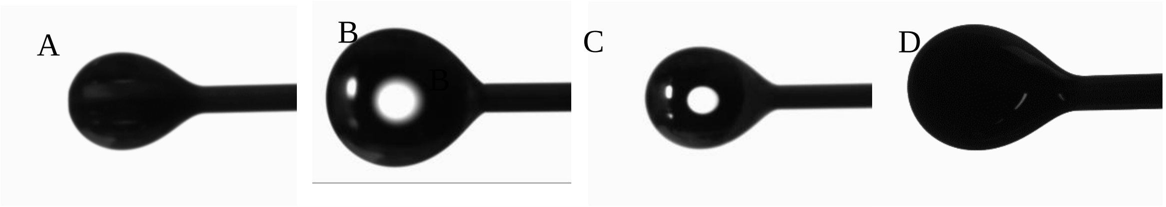


Figure 20: Water (A), DMF (B), crude oil A (C) and crude oil B (D) pendants

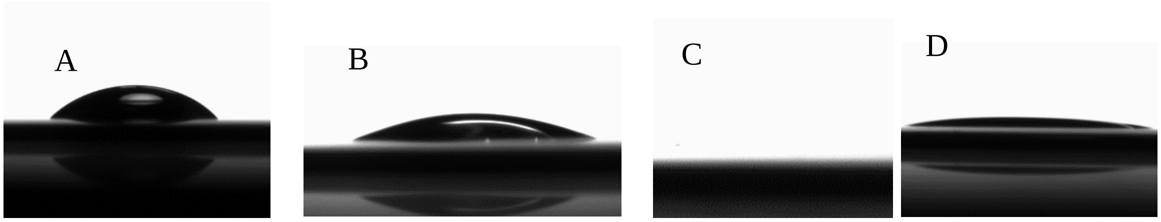


Figure 21: Water (A), DMF (B), crude oil A (C), and crude oil B (D) droplets on silica sensor

In addition, Fourier transform infrared (FTIR) spectroscopy was used to determine the surface composition of the sensor. It was composed of neat SiO_2.

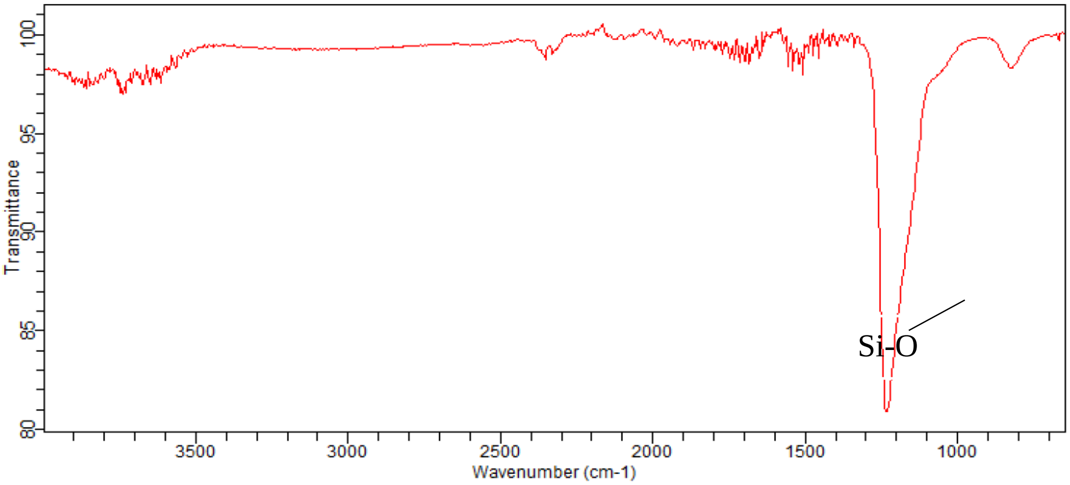


Figure 22: FTIR spectrum of silica sensor

c. Work of adhesion

The work needed for the separation of the interface with the state of equilibrium of liquid-liquid or liquid-solid phase boundary defines the work of adhesion.

The work of adhesion of oil A on the silica surface was also determined by the optical tensiometer to be around 67.14 mN/m and that of oil B on the same surface was 78.81 mN/m.

B. Methodology

1. Preparation of solutions

a. Preparation of surfactant solutions

For the SDS solutions, aliquots of different surfactant concentration were prepared and the amount of SDS in grams was calculated accordingly. The amount measured will be added to a 100 mL beaker and then filled with distilled water to reach the volume needed (100mL).

For example, to prepare 0.1 wt% SDS solution, 0.1 g of the surfactant is needed and the beaker is filled with distilled water to reach 100 mL. Table 5 shows all the SDS solutions used in this research.

Table 5: weight of SDS for different SDS solution samples

Sample no	SDS concentration (wt%)	Amount of SDS (g)	Total volume (mL)
1	0.01	0.0100	100
2	0.05	0.0500	100
3	0.10	0.1000	100
4	0.20	0.2000	100
5	0.50	0.5000	100
6	1.00	1.0000	100
8	2.00	2.0000	100

For the Triton X-100 solutions, a stock solution of 0.04 wt% (0.618 mM) was prepared and different dilutions were performed accordingly.

The stock solution (0.618 mM) was prepared by adding 0.03738 mL of Triton X-100 and filling the beaker with distilled water until reaching 500 mL. The different dilutions are shown in table 6. V_{stock} is the volume that should be taken from the stock solution while V_{DW} is the volume of distilled water that should be added to the solution.

Table 6: The stock volume needed for the different Triton X-100 concentrations

Sample no	Triton X-100 concentration (wt%)	Triton X-100 concentration (mM)	V_{stock} (mL)	V_{DW} (mL)	Total volume (mL)
1	0.0400	0.6180	100.00	0.00	100.00
2	0.0300	0.4637	75.03	24.97	100.00
3	0.0200	0.3090	50.00	50.00	100.00
4	0.0150	0.2318	37.51	62.49	100.00
5	0.0100	0.1546	25.01	74.99	100.00
6	0.0050	0.0773	12.50	87.50	100.00
7	0.0020	0.0309	5.002	94.998	100.00
8	0.0015	0.0232	3.751	96.249	100.00
9	0.0010	0.0155	2.500	97.500	100.00
10	0.0005	0.0077	1.250	98.750	100.00
11	0.0001	0.0015	0.250	99.750	100.00

b. Preparation of brine solutions

Three different brine solutions were prepared in 100 mL flasks. The salt compositions were used for the adsorption process to mimic the reservoir conditions and study the effect of electrolyte in the removal of oil. The solutions were labelled as low salinity (LS) and moderate salinity (MS).The different concentrations are presented in the table below.

Table 7: Amount of NaCl in different concentrations of brine solutions

Solution	Concentration (ppm)	Amount of NaCl (g)	Total volume (mL)
Low Salinity (LS)	2000	0.2	100
Moderate Salinity (MS)	6000	0.6	100

c. Preparation of surfactants in saline solutions

For the low salinity – surfactant solutions and moderate salinity – surfactant solutions, 0.2 g and 0.6 g of NaCl were added respectively with different amount of surfactant to a 100 mL beaker and the rest is filled with distilled water.

For the SDS surfactant, the proper amount of SDS and NaCl is shown in tables 8 and 9 for low and moderate salinity respectively.

Table 8: Amount of SDS in low salinity solutions

SDS concentration in LS (%)	Amount of SDS (g)	Amount of NaCl (g)
0.005	0.0050	0.2000
0.010	0.0100	0.2000
0.020	0.0200	0.2000
0.050	0.0500	0.2000
0.100	0.1000	0.2000
0.200	0.2000	0.2000

Table 9: Amount of SDS in moderately saline solutions

SDS concentration in MS (%)	Amount of SDS (g)	Amount of NaCl (g)
0.002	0.002	0.6000
0.005	0.005	0.6000
0.010	0.010	0.6000
0.050	0.050	0.6000
0.100	0.100	0.6000
0.200	0.200	0.6000

For the Triton X-100 surfactant solutions, the amount of Triton X-100 and NaCl is shown in tables 10 and 11 for low and moderately saline solutions respectively. These solutions are prepared by diluting a solution of 0.04% Triton X-100 in distilled water.

Table 10: Amount of Triton X-100 in low salinity solution

Triton X-100 concentration in LS	Volume from stock solution (0.04% w/v)	Amount of NaCl (g)
0.0001	0.2500	0.2000
0.0005	1.2505	0.2000
0.0010	2.5000	0.2000
0.0020	5.0019	0.2000
0.0050	12.5048	0.2000
0.0100	25.0097	0.2000
0.0150	37.5081	0.2000
0.0200	50.0194	0.2000

Table 11: Amount of Triton X-100 in moderately saline solutions

Triton X-100 concentration in MS	Amount of Triton X-100 (mL)	Amount of NaCl (g)
0.0001	0.2500	0.6000
0.0005	1.2505	0.6000
0.0010	2.5000	0.6000
0.0015	3.7508	0.6000
0.0020	5.0019	0.6000
0.0050	12.5048	0.6000
0.0100	25.0097	0.6000
0.0150	37.5081	0.6000

d. Preparation of silica solution

High concentration of silica was prepared by mixing smashed particles in deionized water. The mixture was stirred and sonicated for 10 min. The suspension was centrifuged for 3 min followed by decantation to remove the sediment.

e. Preparation of crude oil B solution

Different concentrations of crude oil were tested in different solvents to check their miscibility. Afterwards, the appropriate solution was spin coated on the silica sensor and checked for readability using the QCM-D.

2. Coating of sensors

The silica solution prepared in the section above was used to prepare a mineral surface on the gold sensor. Afterwards, the oil studied, whether synthetic or crude, was coated on the silica surface.

3. Contact angle measurement

Using the optical tensiometer, the measurement of the contact angle was considered by dispensing a water droplet on the surface. The droplet volume was measured by a mechanical injector and set to 3 µL. In order to avoid any fluid still existing on the tip, a Teflon syringe was utilized. A CCD camera linked to an image analyzer recorded how the droplet has extended and from the base width and the height, the contact angle was automatically calculated.

4. CMC determination

The CMC for all the surfactants with different salinities was determined. For the anionic surfactant SDS, the CMC was found using the conductivity method, while for the nonionic surfactant, the surface tension using the optical tensiometer was performed.

5. Sensors cleaning

a. Gold sensors

Preparation of the cleaning solution: 10 mL of distilled water, 2 mL of ammonia NH_3 (under the hood) and 2 mL of hydrogen peroxide H_2O_2 are added to a 50 mL beaker.

Heating the cleaning solution: the solution will be heated using an electric heater under the hood until the temperature reaches 80ºC. The heated solution will be poured into the beaker containing the holder holding the sensors.

Drying the sensors: Each sensor should be hold by a tweezer separately for the drying process. Ethanol should be dropped on both surfaces. The sensor should then be put under a gentle flow of filtered dry air or N_2 gas in order to dry them completely. After that, distilled water (control) should be dropped on both surfaces and then, again, put under gentle flow of filtered dry air or N_2 gas. The sensors should be placed in a clean and sterile Petri-Dish.

UV light exposure: the Petri-Dish containing the sensors should be closed and stapled tightly and then placed under UV light in a UV chamber at a 90-degree position (in this way the sensors are normal to the light source).

b. Silica sensors

Treatment with UV light for 10 minutes.

2% SDS in distilled water should be prepared: 2 g of SDS added to 100 mL distilled water.

The solution was poured into the beaker containing the holder holding the sensors and sonicated for 30 minutes at room temperature.

Drying the sensor: Each sensor should be hold by a tweezer separately for the drying process. It is essential to keep the surfaces wet when submerging them with SDS before rinsing them well with distilled water. The sensor should then be put under a gentle flow of filtered dry air or N_2 gas in order to dry them completely.

The sensors should be placed in a clean and sterile Petri-Dish.

UV light exposure: the Petri-Dish containing the sensors should be closed and stapled tightly and then placed under UV light in a UV chamber at a 90-degree position (in this way the sensors are normal to the light source).

CHAPTER III

RESULTS

A. Optimization

1. Crude oil B solution

After spin coating crude oil B on the silica sensor, the initial frequencies were not readable using the QCM-D due to the high thickness of the oil on the surface. Diluting oil B with different solvent was tested: ethanol and acetone were not miscible while hexane was otherwise. After choosing the right solvent, 8 different concentrations of crude oil B were diluted in hexane were tested from 0.100 g/mL to 0.002 g/mL as shown in figure 23.

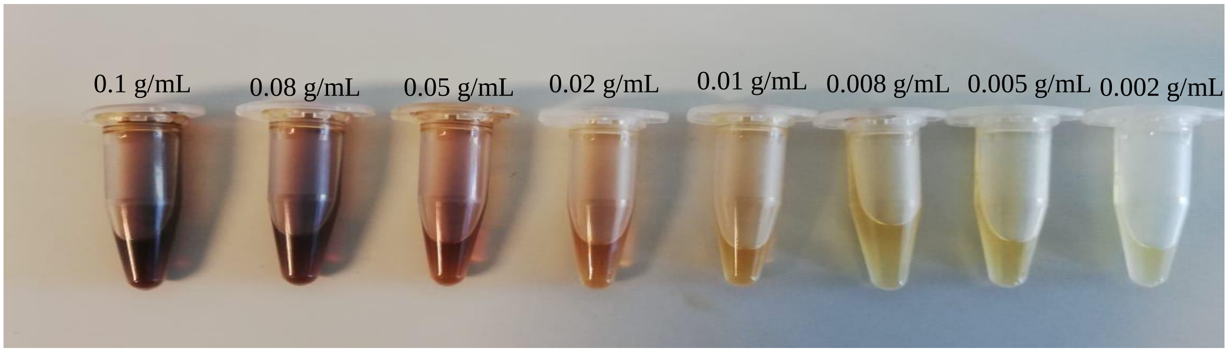
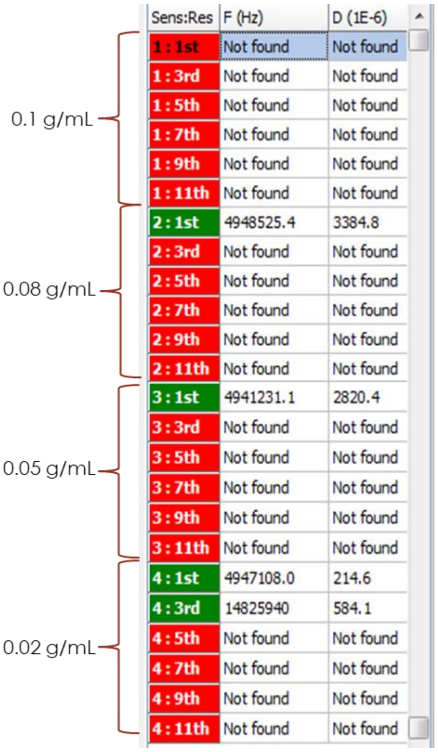
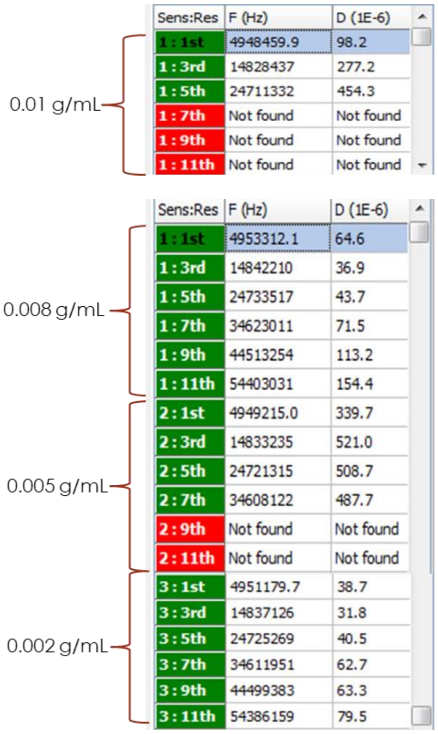


Figure 23: Samples of different concentration of crude oil B in hexane

After filtering the samples with syringe filters (0.22 µm), 20µL of crude oil B solutions were coated on the silica sensors and left for 30 min to dry. The QCM-D showed the following setup measurement results:



Sens:Res	F (Hz)	D (1E-6)
1 : 1st	Not found	Not found
1 : 3rd	Not found	Not found
1 : 5th	Not found	Not found
1 : 7th	Not found	Not found
1 : 9th	Not found	Not found
1 : 11th	Not found	Not found
2 : 1st	4948525.4	3384.8
2 : 3rd	Not found	Not found
2 : 5th	Not found	Not found
2 : 7th	Not found	Not found
2 : 9th	Not found	Not found
2 : 11th	Not found	Not found
3 : 1st	4941231.1	2820.4
3 : 3rd	Not found	Not found
3 : 5th	Not found	Not found
3 : 7th	Not found	Not found
3 : 9th	Not found	Not found
3 : 11th	Not found	Not found
4 : 1st	4947108.0	214.6
4 : 3rd	14825940	584.1
4 : 5th	Not found	Not found
4 : 7th	Not found	Not found
4 : 9th	Not found	Not found
4 : 11th	Not found	Not found



Sens:Res	F (Hz)	D (1E-6)
1 : 1st	4948459.9	98.2
1 : 3rd	14828437	277.2
1 : 5th	24711332	454.3
1 : 7th	Not found	Not found
1 : 9th	Not found	Not found
1 : 11th	Not found	Not found

Sens:Res	F (Hz)	D (1E-6)
1 : 1st	4953312.1	64.6
1 : 3rd	14842210	36.9
1 : 5th	24733517	43.7
1 : 7th	34623011	71.5
1 : 9th	44513254	113.2
1 : 11th	54403031	154.4
2 : 1st	4949215.0	339.7
2 : 3rd	14833235	521.0
2 : 5th	24721315	508.7
2 : 7th	34608122	487.7
2 : 9th	Not found	Not found
2 : 11th	Not found	Not found
3 : 1st	4951179.7	38.7
3 : 3rd	14837126	31.8
3 : 5th	24725269	40.5
3 : 7th	34611951	62.7
3 : 9th	44499383	63.3
3 : 11th	54386159	79.5

From these results, the maximum oil concentration that can be used is 0.02 g/mL in hexane showing the 3rd overtone, which is the overtone studies in this research. The silica sensor coated with this concentration of oil is shown in the figure below.

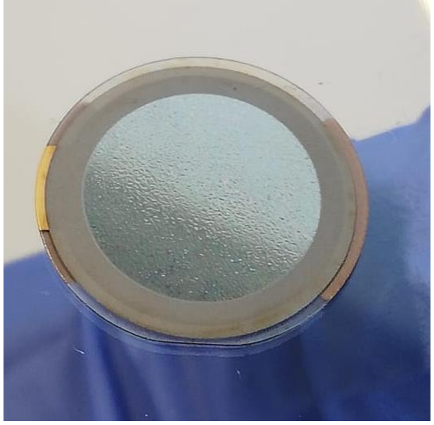
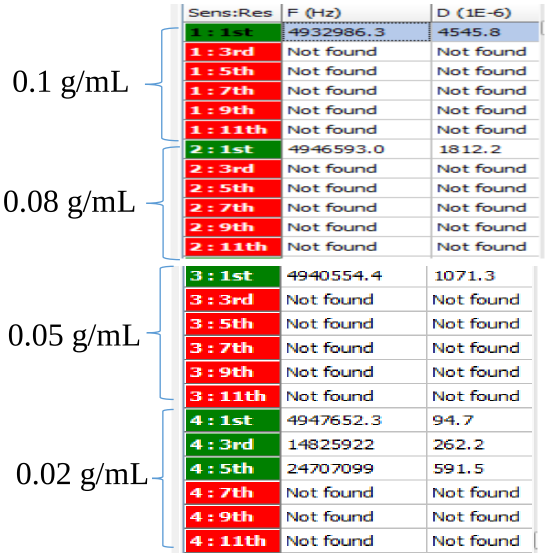


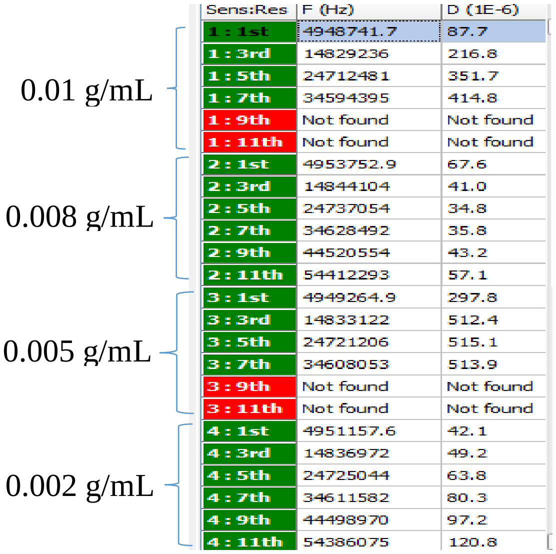
Figure 24: Silica sensor coated with 0.02 g/mL of crude oil B in hexane

The contact angle was then evaluated to check for the hydrophobicity of the surface: the contact angle of water – air – surface has increased from an average of 42° for a clean sensor until reaching an average of 68° for a coated sensor.

After 24h, the sensors were re-evaluated using the QCM-D and the 3rd overtone was shown for a maximum of 0.02 g/mL crude oil B in hexane, which is the concentration adopted in this study:



Sens:Res	F (Hz)	D (1E-6)
1 : 1st	4932986.3	4545.8
1 : 3rd	Not found	Not found
1 : 5th	Not found	Not found
1 : 7th	Not found	Not found
1 : 9th	Not found	Not found
1 : 11th	Not found	Not found
2 : 1st	4946593.0	1812.2
2 : 3rd	Not found	Not found
2 : 5th	Not found	Not found
2 : 7th	Not found	Not found
2 : 9th	Not found	Not found
2 : 11th	Not found	Not found
3 : 1st	4940554.4	1071.3
3 : 3rd	Not found	Not found
3 : 5th	Not found	Not found
3 : 7th	Not found	Not found
3 : 9th	Not found	Not found
3 : 11th	Not found	Not found
4 : 1st	4947652.3	94.7
4 : 3rd	14825922	262.2
4 : 5th	24707099	591.5
4 : 7th	Not found	Not found
4 : 9th	Not found	Not found
4 : 11th	Not found	Not found



Sens:Res	F (Hz)	D (1E-6)
1 : 1st	4948741.7	87.7
1 : 3rd	14829236	216.8
1 : 5th	24712481	351.7
1 : 7th	34594395	414.8
1 : 9th	Not found	Not found
1 : 11th	Not found	Not found
2 : 1st	4953752.9	67.6
2 : 3rd	14844104	41.0
2 : 5th	24737054	34.8
2 : 7th	34628492	35.8
2 : 9th	44520554	43.2
2 : 11th	54412293	57.1
3 : 1st	4949264.9	297.8
3 : 3rd	14833122	512.4
3 : 5th	24721206	515.1
3 : 7th	34608053	513.9
3 : 9th	Not found	Not found
3 : 11th	Not found	Not found
4 : 1st	4951157.6	42.1
4 : 3rd	14836972	49.2
4 : 5th	24725044	63.8
4 : 7th	34611582	80.3
4 : 9th	44498970	97.2
4 : 11th	54386075	120.8

2. *Coating of gold with silica*

For the first trial, 0.5 mL of 0.4 wt% silica suspension was pipetted to the gold sensor placed on the spin coater at a speed of 250 rpm for 30s. Another 0.5 mL of solution were added to the spinning surface at 2000 rpm for 30s and then leaving it for 60s at 5000 rpm. The results were not satisfying since the surface remained hydrophobic even after coating with silica. This is also shown by the poor amount of silica adhered to the surface as shown in the table below.

Table 12: The contact angle and weight of the gold sensor before and after coating with silica for trial 1

		Before coating with silica	**After coating with silica**
Gold sensor 1	CA (°) – average	93.02	84.70
	m (mg)	115.462	115.481
Gold sensor 2	CA (°) – average	85.52	78.52
	m (mg)	114.801	114.838
Gold sensor 3	CA (°) – average	75.1	68.2
	m (mg)	112.595	112.635

The water droplets on the different silica sensors are presented in figure 25, 26 and 27 before and after coating the gold sensors with the silica solutions showing the stabilization of the hydrophobicity.

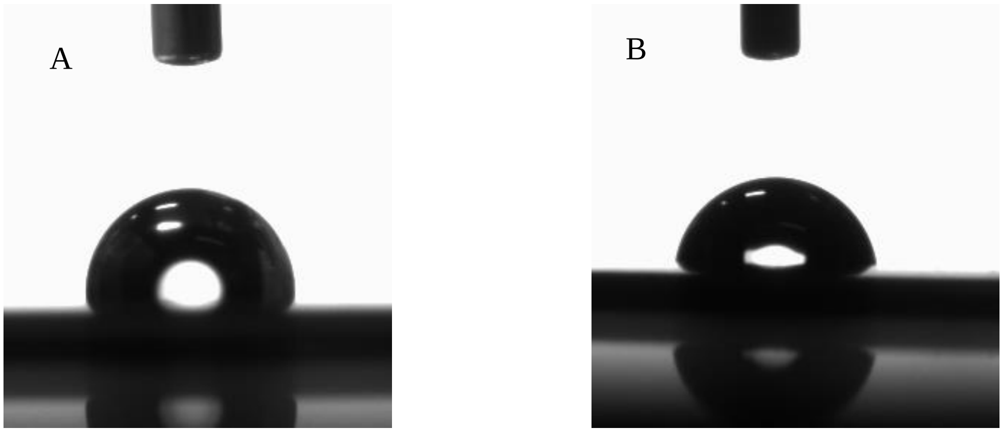


Figure 25: Water droplet on gold sensor 1 before (A) and after (B) coating with silica

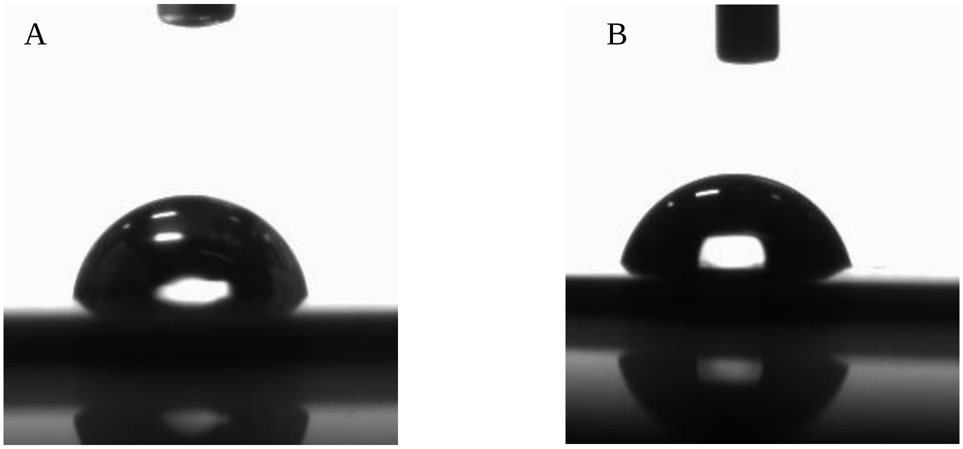


Figure 26: Water droplet on gold sensor 2 before (A) and after (B) coating with silica

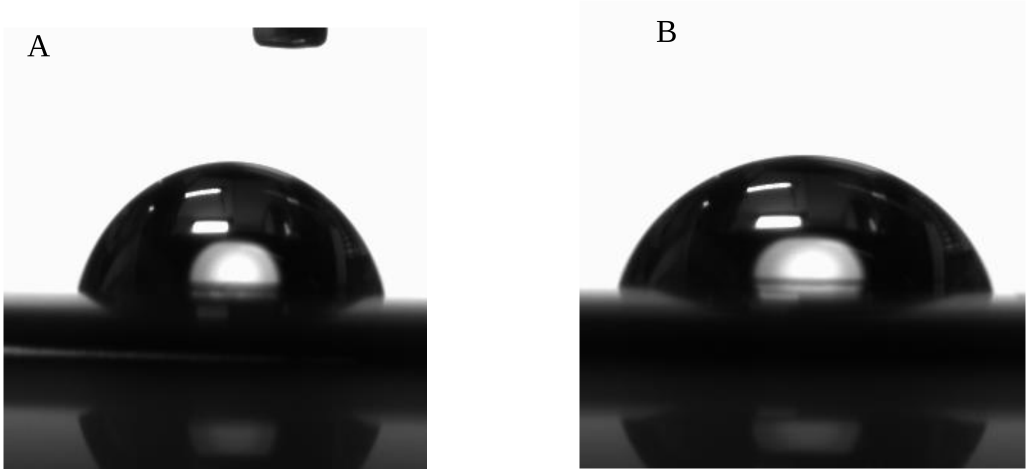


Figure 27: Water droplet on gold sensor before (A) and after (B) coating with silica

After different trials, the following speed and time were programmed to obtain a relatively smooth and uniform silica surface: step1: 100 rpm for 30s; step2: 300 rpm for 30s; step3: 1000 rpm for 60s; step4: 3000 rpm for 60s and step5: 5000 rpm for 30s. The results, shown in table 13, were acceptable with a quite good adhesion of silica on the surface.

Table 13: The contact angle and weight of the gold sensor before and after coating with silica with the appropriate speed and time of spin coating

		Before coating with silica	**After coating with silica**
Gold sensor 1	CA (^{o}) – average	89.92	19.94
	m (mg)	112.037	112.074
Gold sensor 2	CA (^{o}) – average	92.32	29.98
	m (mg)	114.800	114.839
Gold sensor 3	CA (^{o}) – average	83.88	26.23
	m (mg)	113.979	114.022

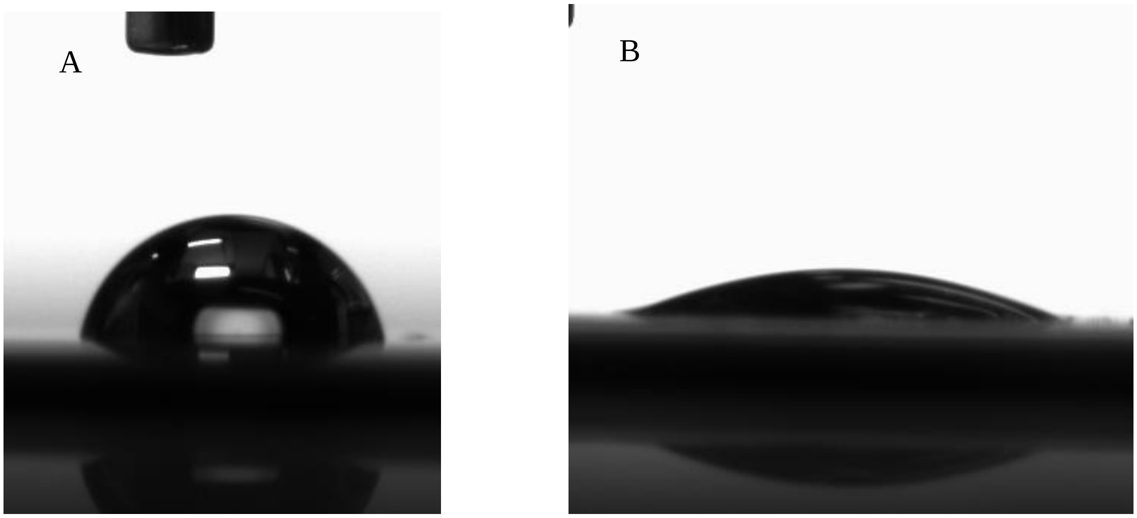


Figure 28: Water droplet on gold sensor 1 before (A) and after (B) coated with silica

To make sure that the silica is well adhered on the surface of the gold sensor, water has been injected until a baseline is perceived. A small amount of silica was removed and the contact angle remained approximately the same as shown in the table below.

Table 14: The contact angle and weight of the silica coated gold sensor before and after injecting the QCM-D with distilled water

		Before coating with silica	**After coating with silica**	**After QCM-D run**
Gold sensor 4	CA (^{o}) – average	76.33	7.62	7.45
	m (mg)	112.609	112.642	112.633
Gold sensor 5	CA (^{o}) – average	77.89	4.46	3.60
	m (mg)	113.995	114.025	114.019

3. *Coating of sensors with dodecane*

Silica coated gold sensors

The silica coated gold sensors where then coated with dodecane. The results for the first trial are shown in the table below:

Table 15: The contact angle and weight of the silica coated sensors before and after coating with dodecane for the first trial

		Before coating with dodecane	**After coating with dodecane**
Silica coated gold sensor 1	CA (^{o}) – average	19.94	24.56
	m (mg)	112.074	112.102
Silica coated gold sensor 2	CA (^{o}) – average	29.98	30.29
	m (mg)	114.839	114.879

After trying different speed and time combination for spin coating, the same results were obtained where the surface of the sensor remained hydrophilic. Hence, dodecane did not adhere on the surface of the silica coated gold sensors.

Table 16: The contact angle and weight of the silica coated sensors before and after coating with dodecane final combination

		Before coating with dodecane	**After coating with dodecane**
Silica coated gold sensor 4	CA (^{o}) – average	7.45	12.82
	m (mg)	112.633	112.640
Silica coated gold sensor 5	CA (^{o}) – average	3.60	8.67
	m (mg)	114.019	114.050

Silica sensors

20 µL of dodecane was pipetted to the silica sensor placed on a spin coater. After different trials, the following speed and time were programmed: step1: 350 rpm for 30s; step2: 550 rpm for 30s; step3: 750 rpm for 30s; step4: 950 rpm for 30s and step5: 1150 rpm for 30s. But the results at different combinations weren't convincing since the contact angle

of the water – air - surface did not increase by much. The different results are presented below:

Table 17: The contact angle and weight of the silica sensors before and after coating with dodecane

		Before coating with dodecane	After coating with dodecane
Silica sensor 1	CA (^{o}) – average	44.46	51.19
	m (mg)	115.117	115.129
Silica sensor 2	CA (^{o}) – average	43.27	50.54
	m (mg)	113.157	113.161
Silica sensor 3	CA (^{o}) – average	36.43	56.87
	m (mg)	113.109	113.141
Silica sensor 4	CA (^{o}) – average	46.28	48.97
	m (mg)	115.116	115.121
Silica sensor 5	CA (^{o}) – average	49.76	48.59
	m (mg)	113.108	113.112

4. *CMC determination*

The CMC is dependent on the molecular structure of the surfactant studied and is subjective to any inorganic salt present in the solution. At the CMC where the formation of micelles start occurring, some characteristics of the surfactant undergo sudden changes such as the conductivity and the surface tension. By studying these changes, the CMC of the two surfactants in three different medium is determined below.

SDS in 0% NaCl (using conductivity method)

Table 18: The conductivity of different SDS concentrations in distilled water.

DW initial volume (mL)	SDS solution added V (mL)	Total V (mL)	Stock SDS (g/L)	SDS solution (g/L)	SDS solution (mM)	Conductivity (uS/cm)
25	0	25	20	0		
25	0.5	25.5	20	0.392	1.36	0.166
25	1	26	20	0.769	2.67	0.232
25	1.5	26.5	20	1.13	3.93	0.308
25	2	27	20	1.48	5.14	0.371
25	2.5	27.5	20	1.82	6.31	0.424
25	3	28	20	2.14	7.44	0.471
25	3.5	28.5	20	2.46	8.53	0.516
25	4	29	20	2.76	9.58	0.554
25	4.5	29.5	20	3.05	10.6	0.588
25	5	30	20	3.33	11.6	0.619
25	5.5	30.5	20	3.61	12.5	0.648
25	6	31	20	3.87	13.4	0.677
25	6.5	31.5	20	4.13	14.3	0.705
25	7	32	20	4.37	15.2	0.731

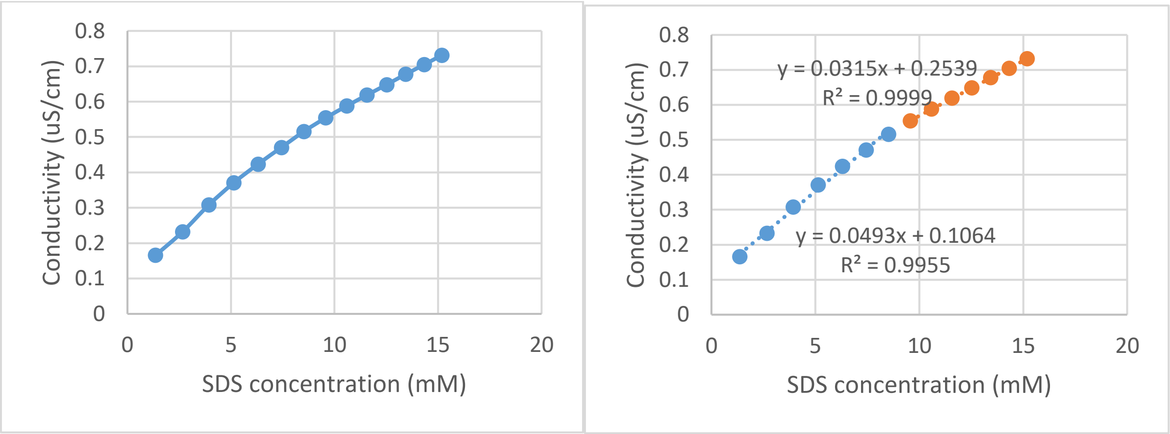


Figure 29: The conductivity vs the SDS concentration in distilled water.

The CMC for the SDS solution in distilled water was confirmed to be 8.29 mM (2.39 g/L).

SDS in 0.2% NaCl (using conductivity method)

Table 19: The conductivity of different SDS concentrations in low salinity solution.

LS initial volume (mL)	SDS solution added V (mL)	Total V (mL)	Stock SDS (g/L)	SDS solution (g/L)	SDS solution (mM)	Conductivity (uS/cm)
25	0	25	15	0		
25	0.5	25.5	15	0.294	1.02	3.73
25	1	26	15	0.577	2.00	3.78
25	1.5	26.5	15	0.849	2.95	3.83
25	2	27	15	1.11	3.86	3.85
25	2.5	27.5	15	1.36	4.73	3.87
25	3	28	15	1.61	5.58	3.89
25	3.5	28.5	15	1.84	6.40	3.91
25	4	29	15	2.07	7.19	3.92
25	4.5	29.5	15	2.29	7.95	3.94
25	5	30	15	2.50	8.68	3.96

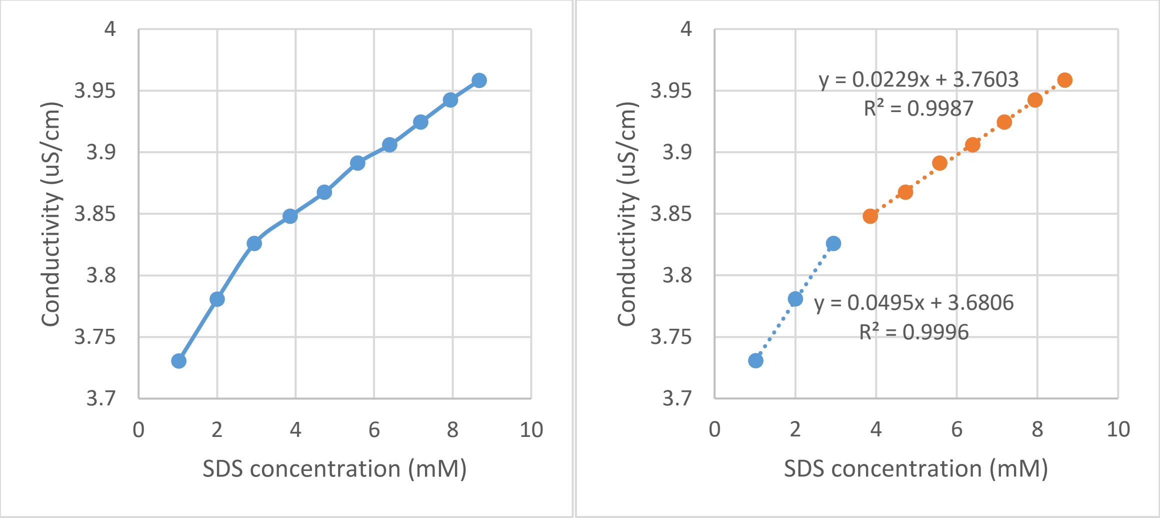


Figure 30: The conductivity vs the SDS concentration in low salinity.

The CMC for the SDS solution in low salinity was determined to be 2.99 mM (0.86 g/L).

SDS in 0.6% NaCl (using conductivity method)

Table 20: The conductivity of different SDS concentration in moderate salinity.

MS initial volume (mL)	SDS solution added V (mL)	Total V (mL)	Stock SDS (g/L)	SDS solution (g/L)	SDS solution (mM)	Conductivity (uS/cm)
40	0	40	15	0		9.99
40	0.5	40.5	15	0.185	0.643	9.98
40	1	41	15	0.366	1.27	10.01
40	1.5	41.5	15	0.542	1.88	10.03
40	2	42	15	0.714	2.48	10.04
40	2.5	42.5	15	0.882	3.06	10.06
40	3	43	15	1.05	3.63	10.07

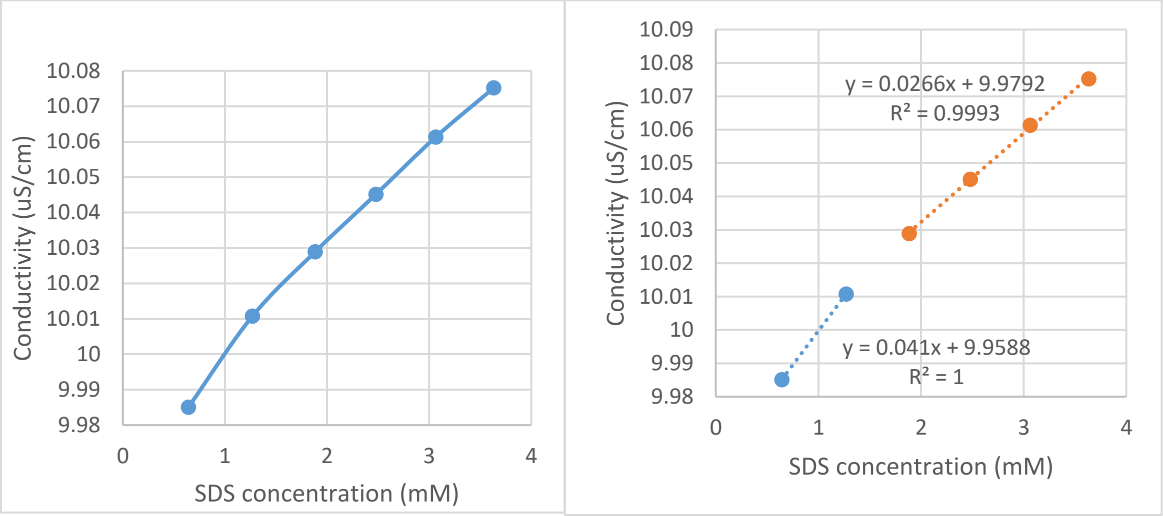


Figure 31: The conductivity vs the SDS conductivity in moderate salinity.

The CMC for the SDS solution in moderate salinity was determined to be 1.4 mM (0.403 g/L).

Triton X-100 in 0% NaCl (using surface tension method)

Table 21: The surface tension of different Triton X-100 concentrations in distilled water.

DW initial volume (mL)	TX solution added V (mL)	Total V (mL)	Stock TX (g/L)	TX solution (g/L)	TX solution (mM)	IFT (mN/m)
25	0	25	0.4	0		
25	0.5	25.5	0.4	0.00784	0.0272	64.5
25	1	26	0.4	0.0154	0.0534	53.9
25	1.5	26.5	0.4	0.0226	0.0786	51.2
25	2	27	0.4	0.0296	0.103	49.3
25	2.5	27.5	0.4	0.0364	0.126	46.8
25	3	28	0.4	0.0429	0.149	46.0
25	3.5	28.5	0.4	0.0491	0.171	43.8
25	4	29	0.4	0.0552	0.192	46.1
25	4.5	29.5	0.4	0.0610	0.212	43.7
25	5	30	0.4	0.0667	0.231	41.1
25	5.5	30.5	0.4	0.0721	0.250	40.4
25	6	31	0.4	0.0774	0.269	39.7
25	6.5	31.5	0.4	0.0825	0.287	38.7
25	7	32	0.4	0.0875	0.304	39.4

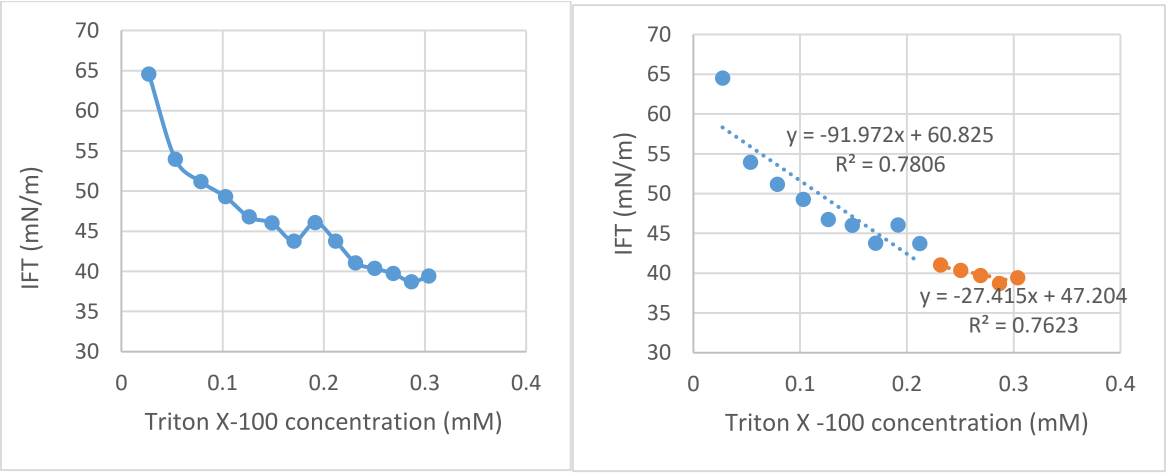


Figure 32: The interfacial tension vs the concentration of Triton X-100 in distilled water

The CMC for the Triton X-100 solution in distilled water was confirmed to be 0.211 mM (0.136 g/L).

Triton X-100 in 0.2% NaCl (using surface tension method)

Table 22: The surface tension of different concentrations of Triton X-100 in low salinity.

DW initial volume (mL)	TX solution added V (mL)	Total V (mL)	Stock TX (g/L)	TX solution (g/L)	TX solution (mM)	IFT (mN/m)
15	0	15	0.4	0		
15	0.5	15.5	0.4	0.0129	0.0199	53.0
15	1	16	0.4	0.0250	0.0386	52.1
15	1.5	16.5	0.4	0.0364	0.0562	51.7
15	2	17	0.4	0.0471	0.0727	46.5
15	2.5	17.5	0.4	0.0571	0.0883	44.3
15	3	18	0.4	0.0667	0.103	42.0
15	3.5	18.5	0.4	0.0757	0.117	37.8
15	4	19	0.4	0.0842	0.130	39.4
15	4.5	19.5	0.4	0.0923	0.143	37.4
15	5	20	0.4	0.100	0.155	37.3

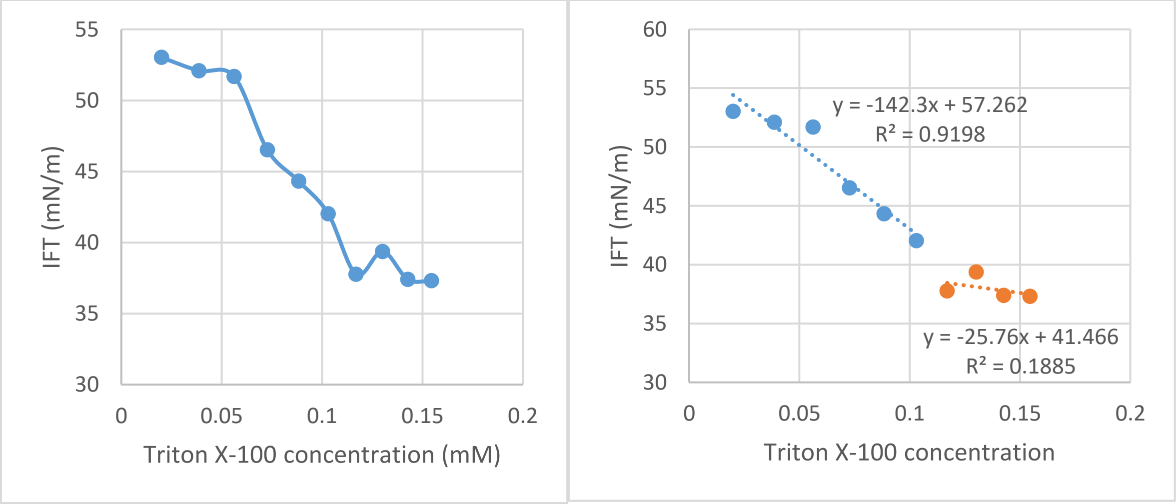


Figure 33: The interfacial tension vs the concentration of Triton X-100 in low salinity.

The CMC for the Triton X-100 solution in low salinity was determined to be 0.135 mM (0.087g/L).

Triton X-100 in 0.6% NaCl (using surface tension method)

Table 23: The surface tension of different Triton X-100 concentrations in moderate salinity.

DW initial volume (mL)	TX solution added V (mL)	Total V (mL)	Stock TX (g/L)	TX solution (g/L)	TX solution (mM)	IFT (mN/m)
15	0	15	0.4	0		
15	0.5	15.5	0.4	0.012903	0.019943	58.28
15	1	16	0.4	0.025	0.03864	50.34
15	1.5	16.5	0.4	0.036364	0.056203	46.72
15	2	17	0.4	0.047059	0.072734	40.79
15	2.5	17.5	0.4	0.057143	0.08832	40.56
15	3	18	0.4	0.066667	0.10304	39.83
15	3.5	18.5	0.4	0.075676	0.116964	40.11

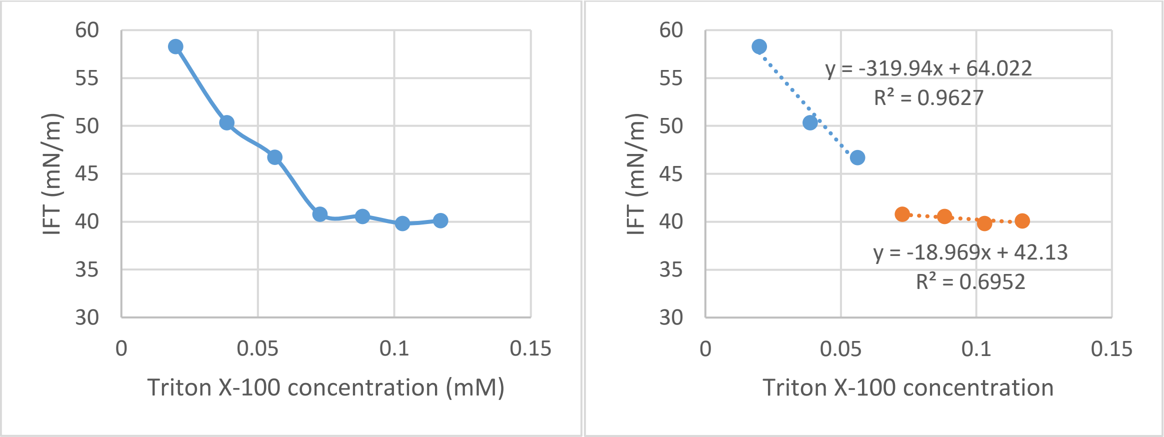


Figure 34: The interfacial tension vs the concentration of Triton X-100 in moderate salinity.

The CMC for the Triton X-100 solution in moderate salinity was determined to be 0.0727 mM (0.047g/L).

B. Crude oil B

For the heavier oil – crude oil B – two surfactants were studied: SDS and Triton X-100. Each of these two surfactants was analyzed with 3 different salinity concentrations: distilled water with is 0% saline, low salinity (0.2% saline) and moderate salinity (0.6% saline). Moreover, with each of these salinity levels, different concentrations of solutions were studied. After different runs, the results that converge are the ones selected and shown in the tables below.

For each concentration of a certain surfactant in a specific salinity, the weight of the sensor before and after coating was determined in order to see how much oil has adsorbed on the surface in mg.

In addition, the initial frequency, the frequency after coating, the frequency before and after desorption were computed in order to check the amount of oil adsorbed and desorbed from the silica surface in mg/cm^2 using Sauebrey's equation. In this way, the percentage of oil desorbed can be estimated.

The dissipation before and after coating of the silica sensor with the crude oil B solution was also computed. The increase in the dissipation indicate the development of a soft molecular film.

Each surfactant concentration in a certain salinity was run separately in order to determine how much this specific amount has desorbed oil on the surface.

All of the runs were performed at a constant temperature of 20ºC and a flow rate of 100 µL/min.

One representative graph is presented for each case, the remaining two graphs will be shown in Appendix A.

1. *Control solutions*

Distilled water (0.0% NaCl)

Table 24: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for distilled water control solution.

	W $_{clean}$ (mg)	W $_{coated}$ (mg)	Adsorption (mg)
Run1	113.535	113.851	0.316
Run2	116.189	116.449	0.260
Run3	115.714	115.918	0.204

For the distilled water control solution, the average of oil adsorbed on the silica surface is 0.260 ± 0.0005 mg.

Table 25: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for the distilled water control solution.

	F $_{clean}$ (Hz)	F $_{coated}$ (Hz)	F $_{before\ desorption}$ (Hz)	F $_{after\ desorption}$ (Hz)	ΔF $_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,847,740	14,836,023	14,835,832	14,843,913	8,081	0.0691	0.0477	0.690
2	14,841,740	14,824,268	14,824,265	14,834,943	10,678	0.103	0.0630	0.611
3	14,837,862	14,819,391	14,819,321	14,832,032	12,711	0.109	0.0750	0.688

The average ratio of oil desorbed from the surface is 0.663 ± 0.0450**.**

Table 26: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for the distilled water control solution.

	D $_{clean}$ (e-6)	D $_{coated}$ (e-6)	ΔD $_{adsorption}$ (e-6)
Run1	15.0	116.2	101.2
Run2	15.1	287.1	272.0
Run3	15.1	658.0	642.9

The average dissipation increase after coating is around 338.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

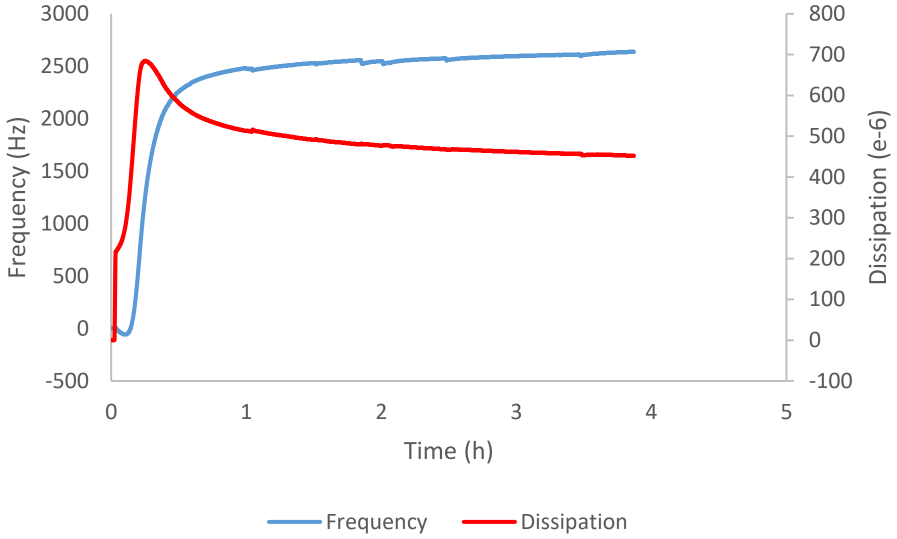


Figure 35: A representative plot showing the change in frequency and dissipation versus time the distilled water control solution

Low Salinity solution (0.2% NaCl)

Table 27: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for the low salinity control solution.

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run 1	115.920	116.000	0.08
Run 2	116.187	116.462	0.275
Run 3	114.088	114.308	0.22

For the low salinity control solution, the average of oil adsorbed on the silica surface is 0.192 ± 0.0005 mg.

Table 28: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for the low salinity control solution.

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,861,931	14,859,622	14,858,032	14,859,456	1,424	0.0136	0.00840	0.617
2	14,842,094	14,831,076	14,831,034	14,838,448	7,414	0.0650	0.0437	0.673
3	14,853,788	14,832,042	14,831,953	14,847,752	15,799	0.128	0.0932	0.727

The average ratio of oil desorbed from the surface is 0.672 ± 0.0550.

Table 29: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for the low salinity control solution.

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run 1	15.8	124.5	108.7
Run 2	25.9	912	886.1
Run 3	16.6	473.1	456.5

The average dissipation increase after coating is around 483.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

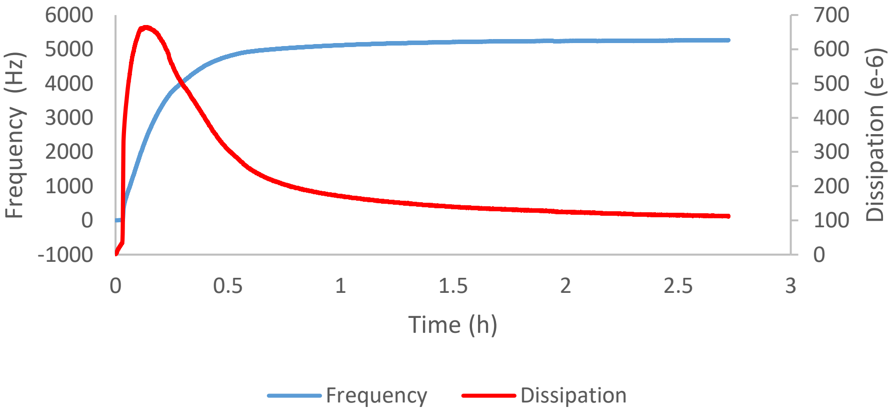


Figure 36: A representative plot showing the change in frequency and dissipation versus time the low salinity control solution

Moderate Salinity solution (0.6% NaCl)

Table 30: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for the moderate salinity control solution

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run 1	116.207	116.625	0.418
Run 2	115.597	115.853	0.256
Run 3	114.768	115.049	0.281

For the distilled water control solution, the average of oil adsorbed on the silica surface is 0.318 ± 0.0005 mg.

Table 31: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for the moderate salinity control solution.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,841,874	14,819,867	14,819,868	14,839,199	19,331	0.130	0.114	0.878
2	14,839,969	14,818,178	14,818,147	14,836,414	18,267	0.129	0.108	0.838
3	14,839,917	14,820,886	14,820,874	14,835,837	14,963	0.112	0.0883	0.786

The average ratio of oil desorbed from the surface is 0.834 ± 0.0461.

Table 32: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for the moderate salinity control solution

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run 1	16.0	1094.0	1078.0
Run 2	25.2	1047.1	1021.9
Run 3	16.0	552.5	536.5

The average dissipation increase after coating is around 878.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

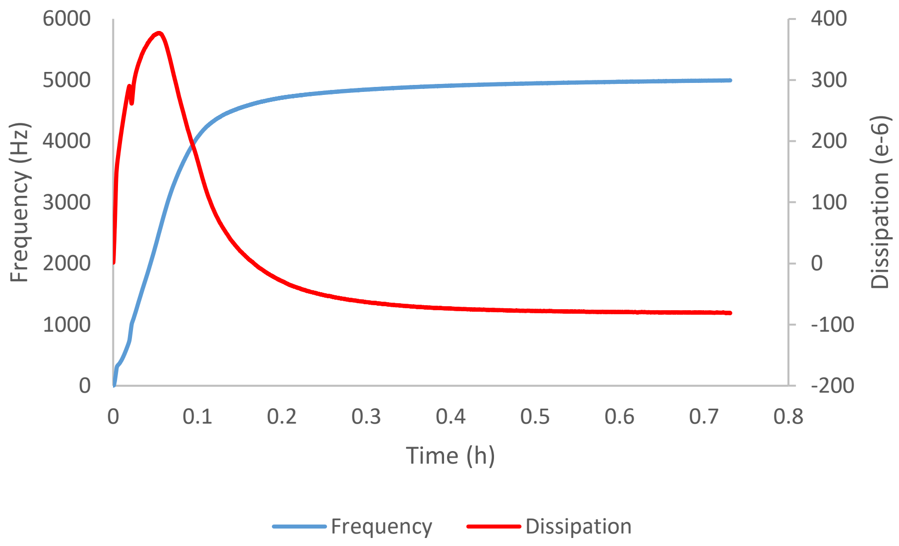


Figure 37: A representative plot showing the change in frequency and dissipation versus time the moderate salinity control solution

2. SDS

a. <u>0% NaCl</u>

0.05% SDS

Table 33: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.05% SDS in distilled water.

	W $_{\text{clean}}$ (mg)	**W $_{\text{coated}}$ (mg)**	**Adsorption (mg)**
Run1	115.802	116.090	0.288
Run2	115.905	116.175	0.270
Run3	113.529	113.633	0.104

For 0.05% SDS in distilled water, the average of oil adsorbed on the surface is 0.221 ± 0.0005 mg.

Table 34: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.05% SDS in distilled water.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,846,482	14,827,497	14,827,134	14,840,254	13,120	0.112	0.0774	0.691
2	14,847,291	14,837,292	14,833,713	14,840,348	6,635	0.0590	0.0391	0.664
3	14,848,078	14,834,781	14,834,508	14,842,944	8,436	0.0785	0.0498	0.634

The average ratio of oil desorbed from the surface is 0.663 ± 0.0285.

Table 35: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.05% SDS in distilled water.

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	25.2	812.6	787.4
Run2	16.2	704.9	688.7
Run3	18.6	178.1	159.5

The average dissipation increase after coating is around 545.2 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

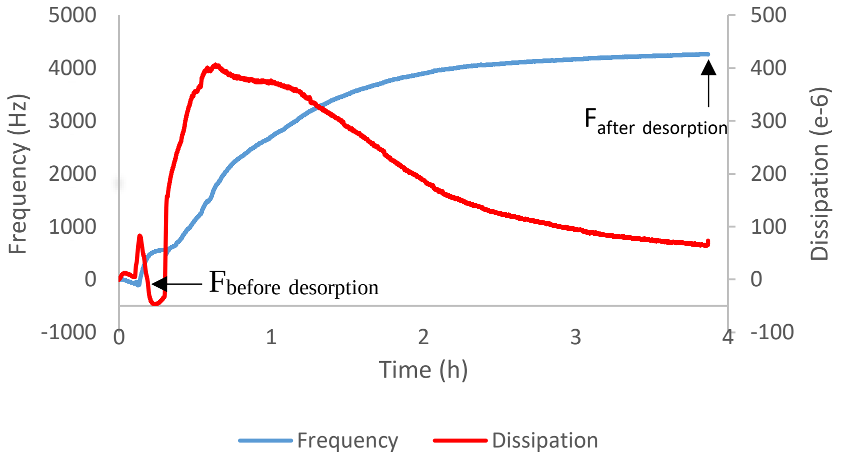


Figure 38: A representative plot showing the change in frequency and dissipation versus time for 0.05% SDS in distilled water.

0.10% SDS

Table 36: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.10% SDS in distilled water.

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	115.125	115.368	0.243
Run2	113.115	113.325	0.210
Run3	115.715	115.963	0.248

For 0.10% SDS in distilled water, the average of oil adsorbed on the surface is 0.234 ± 0.0005 mg.

Table 37:The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.10% SDS in distilled water.

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,201	14,827,546	14,826,847	14,836,926	10,079	0.0688	0.0595	0.865
2	14,846,171	14,830,583	14,830,566	14,844,567	14,001	0.0920	0.0826	0.898
3	14,837,945	14,818,337	14,817,183	14,834,123	16,940	0.116	0.100	0.864

The average ratio of oil desorbed from the surface is 0.876% ± 0.0193.

Table 38: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.10% SDS in distilled water.

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	14.9	893.4	878.5
Run2	16.6	651.9	635.3
Run3	16.1	891.3	875.2

The average dissipation increase after coating is around 796.3 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

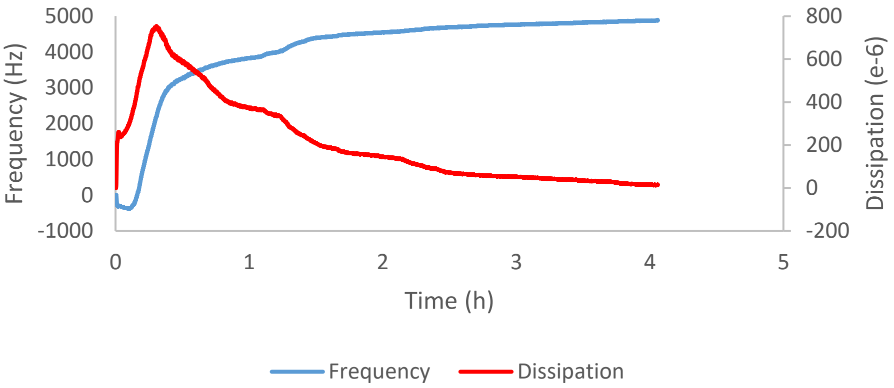


Figure 39:A representative plot showing the change in frequency and dissipation versus time for 0.10% SDS in distilled water.

0.2% SDS

Table 39: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.20% SDS in distilled water.

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	116.211	116.571	0.360
Run2	113.107	113.461	0.354
Run3	116.230	116.478	0.248

For 0.20% SDS in distilled water, the average of oil adsorbed on the surface is 0.321 ± 0.0005 mg.

Table 40: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.20% SDS in distilled water

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,841,429	14,819,565	14,819,411	14,839,775	20,364	0.129	0.120	0.931
2	14,846,086	14,824,379	14,824,326	14,844,261	19,935	0.128	0.118	0.918
3	14,841,186	14,818,121	14,817,733	14,839,928	22,195	0.136	0.131	0.962

The average ratio of oil desorbed from the surface is 0.937 ± 0.0226.

Table 41: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.20% SDS in distilled water

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	16.2	779.0	762.8
Run2	16.0	485.2	469.2
Run3	15.5	735.2	719.7

The average dissipation increase after coating is around 650.6 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

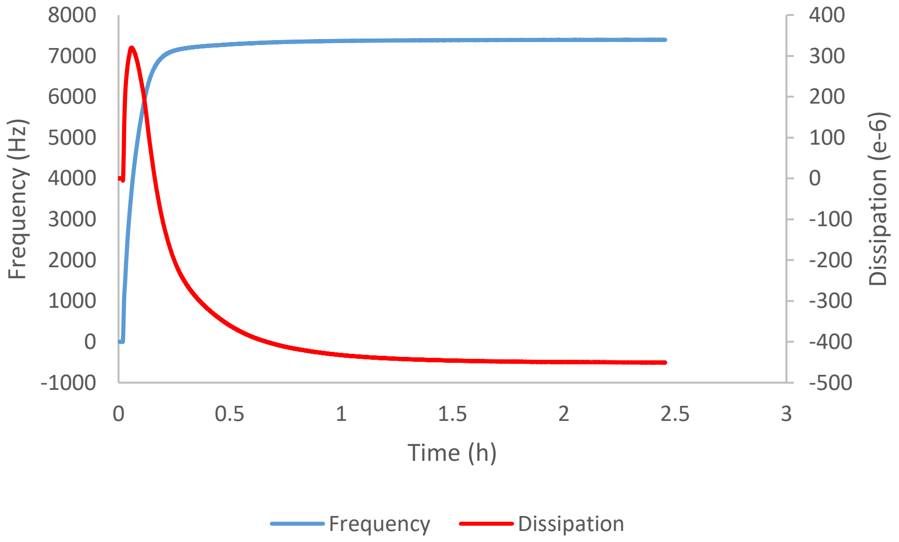


Figure 40: A representative plot showing the change in frequency and dissipation versus time for 0.20% SDS in distilled water.

0.5% SDS

Table 42: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.50% SDS in distilled water.

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run1	116.069	116.453	0.384
Run2	115.120	115.552	0.432
Run3	115.956	116.294	0.338

For 0.50% SDS in distilled water, the average of oil adsorbed on the surface is 0.385 ± 0.0005 mg.

Table 43: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.50% SDS in distilled water

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,841,392	14,816,956	14,816,268	14,839,409	23,141	0.144	0.137	0.947
2	14,839,179	14,813,098	14,812,812	14,837,561	24,749	0.154	0.146	0.949
3	14,861,475	14,845,092	14,844,687	14,860,102	15,415	0.0967	0.0909	0.941

The average ratio of oil desorbed from the surface is 0.946 ± 0.00416.

Table 44: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.50% SDS in distilled water

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	16.7	1060.9	1044.2
Run2	15.2	964.9	949.7
Run3	15.2	974.1	958.9

The average dissipation increase after coating is around 984.3 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

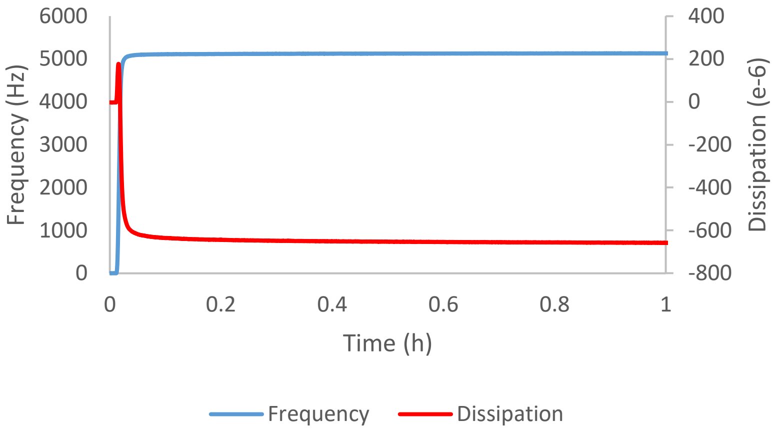


Figure 41: A representative plot showing the change in frequency and dissipation versus time for 0.50% SDS in distilled water.

1.00% SDS

Table 45: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 1.00 % SDS in distilled water

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	114.613	114.934	0.321
Run2	114.775	115.135	0.360
Run3	116.106	116.487	0.381

For 1.00% SDS in distilled water, the average of oil adsorbed on the surface is 0.354 ± 0.0005 mg.

Table 46:The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 1.00% SDS in distilled water

	F$_{clean}$ **(Hz)**	**F**$_{coated}$ **(Hz)**	**F**$_{before\ desorption}$ **(Hz)**	**F**$_{after\ desorption}$ **(Hz)**	**ΔF**$_{desorbed}$ **(Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,840,507	14,822,791	14,822,781	14,839,294	16,513	0.105	0.0974	0.932
2	14,839,651	14,819,307	14,819,369	14,838,548	19,179	0.120	0.113	0.943
3	14,842,517	14,824,906	14,824,905	14,841,154	16,249	0.104	0.0959	0.923

The average ratio of oil desorbed from the surface is 0.932 ± 0.0100.

Table 47: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 1.00% SDS in distilled water

	D$_{clean}$ **(e-6)**	**D**$_{coated}$ **(e-6)**	**ΔD**$_{adsorption}$ **(e-6)**
Run1	15.6	890.7	875.1
Run2	27.5	993.5	966.0
Run3	15.0	702.3	687.3

The average dissipation increase after coating is around 842.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

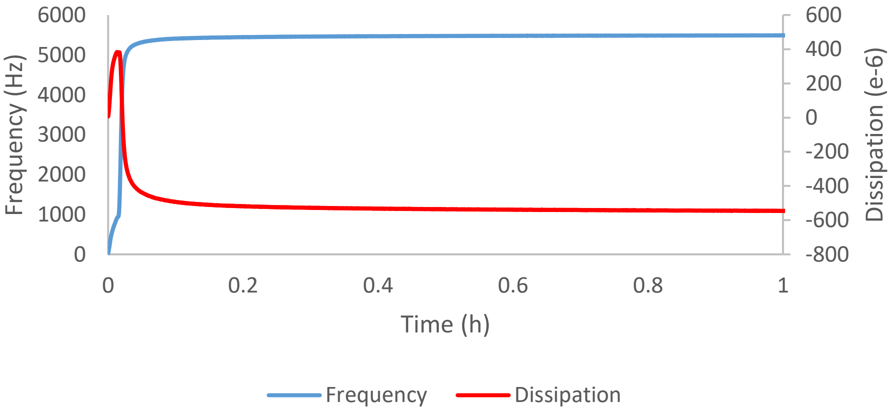


Figure 42: A representative plot showing the change in frequency and dissipation versus time for 1.00% SDS in distilled water.

2.0% SDS

Table 48: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 2.00 % SDS in distilled water

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	113.130	113.385	0.255
Run2	113.406	113.701	0.295
Run3	115.705	116.052	0.347

For 2.00% SDS in distilled water, the average of oil adsorbed on the surface is 0.299 ± 0.0005 mg.

Table 49: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 2.00% SDS in distilled water

	F $_{clean}$ (Hz)	**F $_{coated}$ (Hz)**	**F $_{before\ desorption}$ (Hz)**	**F $_{after\ desorption}$ (Hz)**	**ΔF $_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,846,395	14,822,231	14,822,204	14,845,128	22,924	0.143	0.135	0.949
2	14,850,048	14,830,649	14,830,634	14,848,289	17,655	0.114	0.104	0.910
3	14,838,384	14,818,609	14,818,580	14,836,809	18,229	0.117	0.108	0.922

The average ratio of oil desorbed from the surface is 0.927 ± 0.0200.

Table 50: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 2.00% SDS in distilled water

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	16.7	1058.6	1041.9
Run2	189.5	944.5	755.0
Run3	14.9	989.3	974.4

The average dissipation increase after coating is around 923.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

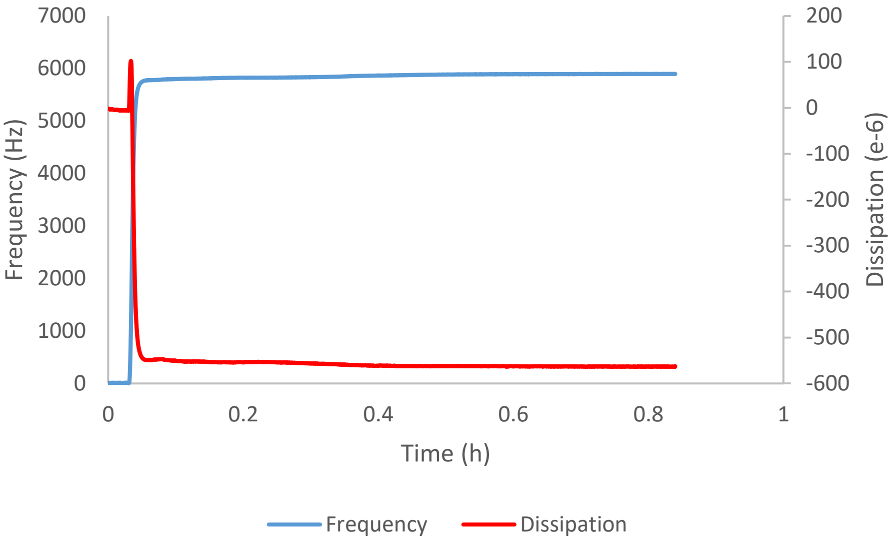


Figure 43: A representative plot showing the change in frequency and dissipation versus time for 2.00% SDS in distilled water.

b. <u>0.2% NaCl</u>

0.005% SDS

Table 51: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.005 % SDS in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	115.714	115.985	0.271
Run2	115.778	116.044	0.266
Run3	112.949	113.299	0.350

For 0.005% SDS in low salinity, the average of oil adsorbed on the surface is 0.296 ± 0.0005 mg.

Table 52: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.005% SDS in low salinity

	F clean (Hz)	**F coated (Hz)**	**F before desorption (Hz)**	**F after desorption (Hz)**	**ΔF desorbed (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,837,788	14,821,693	14,821,133	14,831,807	10,674	0.0950	0.0630	0.663
2	14,862,896	14,841,576	14,841,364	14,855,980	14,616	0.126	0.0862	0.686
3	14,845,179	14,833,317	14,833,088	14,843,653	10,565	0.0700	0.0623	0.725

The average ratio of oil desorbed from the surface is 0.691 ± 0.0313.

Table 53: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.005% SDS in low salinity

	D clean (e-6)	**D coated (e-6)**	**ΔD adsorption (e-6)**
Run1	14.9	612.5	597.6
Run2	16.1	956.1	940.0
Run3	16.7	808.9	792.2

The average dissipation increase after coating is around 776.6 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

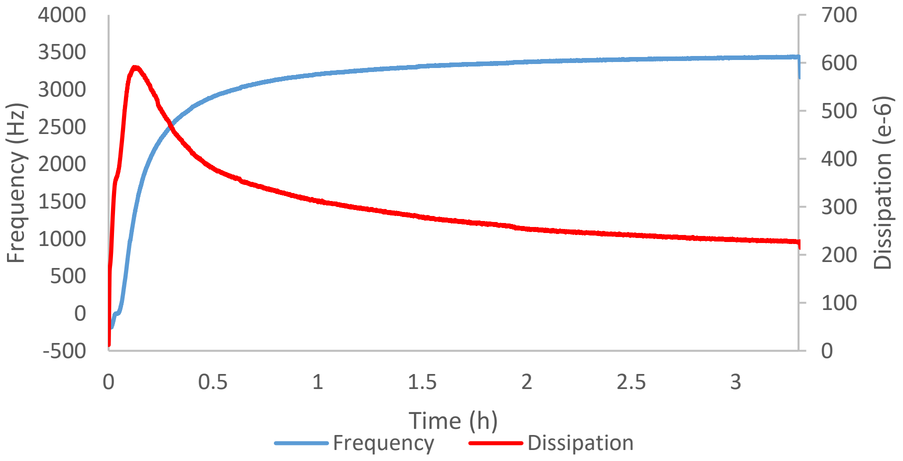


Figure 44: A representative plot showing the change in frequency and dissipation versus time for 0.005% SDS in low salinity.

0.010% SDS

Table 54: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.010 % SDS in low salinity

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	113.529	113.826	0.297
Run2	116.105	116.523	0.418
Run3	116.209	116.574	0.365

For 0.010% SDS in low salinity, the average of oil adsorbed on the surface is 0.360 ± 0.0005 mg.

Table 55: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.010% SDS in low salinity

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,847,755	14,827,357	14,827,241	14,844,077	16,836	0.120	0.0993	0.825
2	14,841,948	14,821,036	14,821,045	14,839,209	18,164	0.123	0.107	0.869
3	14,842,048	14,823,389	14,823,265	14,839,519	16,254	0.110	0.0959	0.871

The average ratio of oil desorbed from the surface is 0.855 ± 0.0260.

Table 56: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.010% SDS in low salinity

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.0	682.0	667.0
Run2	15.3	1025.5	1010.2
Run3	15.2	973.4	958.2

The average dissipation increase after coating is around 878.5 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

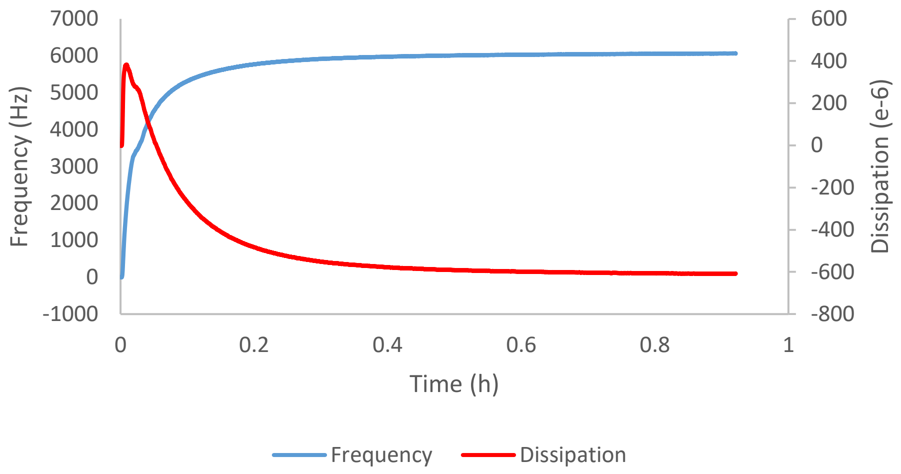


Figure 45: A representative plot showing the change in frequency and dissipation versus time for 0.010% SDS in low salinity.

0.020% SDS

Table 57: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.020 % SDS in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.219	116.441	0.222
Run2	116.217	116.541	0.324
Run3	114.093	114.448	0.355

For 0.020% SDS in low salinity, the average of oil adsorbed on the surface is 0.300 ± 0.0005 mg.

Table 58: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.020% SDS in low salinity

	F clean (Hz)	**F coated (Hz)**	**F before desorption (Hz)**	**F after desorption (Hz)**	**ΔF desorbed (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,841,373	14,818,532	14,818,508	14,839,837	21,329	0.135	0.126	0.934
2	14,841,598	14,813,828	14,813,638	14,838,183	24,545	0.164	0.145	0.884
3	14,854,192	14,832,055	14,832,066	14,852,650	20,584	0.131	0.121	0.930

The average ratio of oil desorbed from the surface is 0.916 ± 0.0278%.

Table 59: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.020% SDS in low salinity

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	16.2	896.5	880.3
Run2	19.1	1109.8	1090.7
Run3	18.5	1025.1	1006.6

The average dissipation increase after coating is around 992.5 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

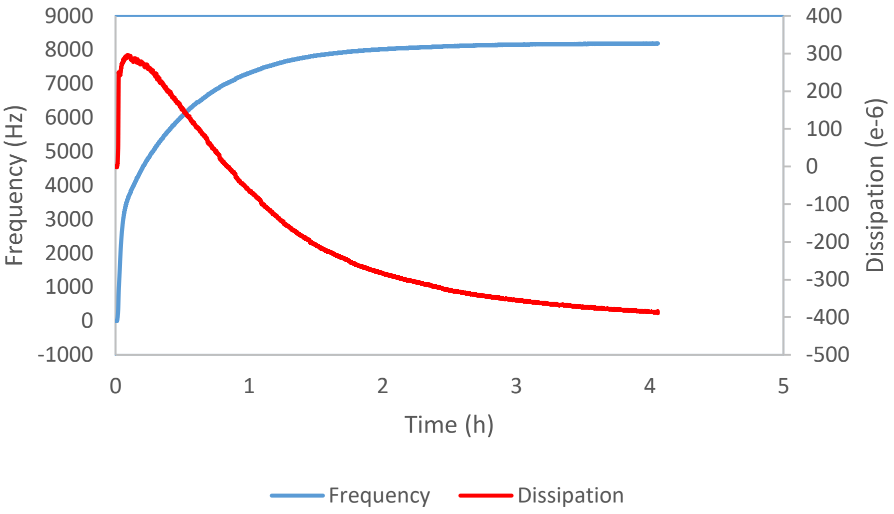


Figure 46: A representative plot showing the change in frequency and dissipation versus time for 0.020% SDS in low salinity.

0.050% SDS

Table 60: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.050 % SDS in low salinity

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	113.126	113.381	0.255
Run2	115.780	116.073	0.293
Run3	113.116	113.447	0.331

For 0.050% SDS in low salinity, the average of oil adsorbed on the surface is 0.293 ± 0.0005 mg.

Table 61: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.050% SDS in low salinity

	F_{clean} (Hz)	**F_{coated} (Hz)**	**$F_{before\ desorption}$ (Hz)**	**$F_{after\ desorption}$ (Hz)**	**$\Delta F_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,846,624	14,825,617	14,825,403	14,844,751	19,348	0.124	0.114	0.921
2	14,863,114	14,841,898	14,841,895	14,861,609	19,714	0.125	0.116	0.929
3	14,846,023	14,820,609	14,820,633	14,844,799	24,166	0.150	0.143	0.951

The average ratio of oil desorbed from the surface is 0.934 ± 0.0155.

Table 62: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.050% SDS in low salinity

	D_{clean} (e-6)	**D_{coated} (e-6)**	**$\Delta D_{adsorption}$ (e-6)**
Run1	18.1	585.8	567.7
Run2	21.2	200.4	179.2
Run3	20.1	1060.3	1040.2

The average dissipation increase after coating is around 595.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

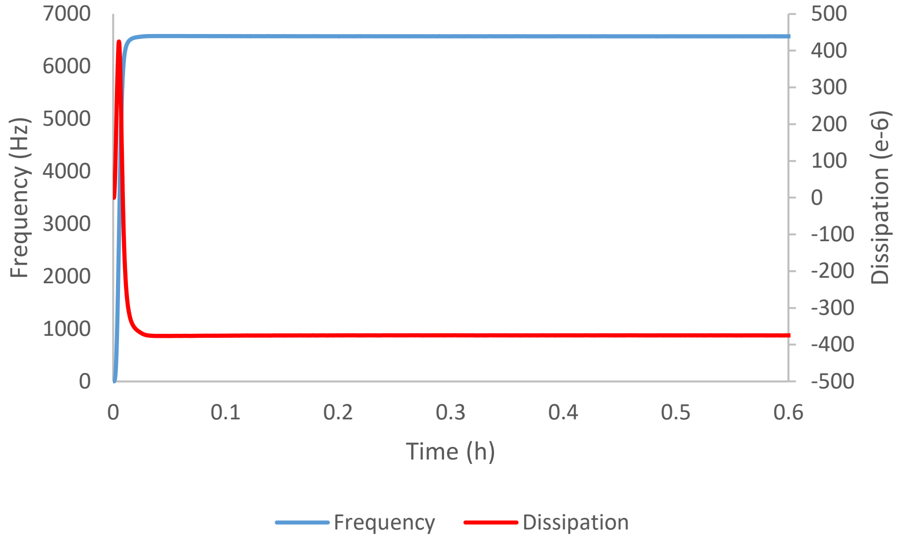


Figure 47: A representative plot showing the change in frequency and dissipation versus time for 0.050% SDS in low salinity

0.100% SDS

Table 63: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.100 % SDS in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.983	117.283	0.300
Run2	115.623	115.850	0.227
Run3	114.767	115.057	0.290

For 0.005% SDS in low salinity, the average of oil adsorbed on the surface is 0.272 ± 0.0005 mg.

Table 64: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.100% SDS in low salinity

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,827,964	14,803,064	14,803,010	14,826,292	23,282	0.147	0.137	0.935
2	14,839,449	14,831,353	14,830,499	14,838,100	7,601	0.0478	0.0448	0.939
3	14,839,878	14,818,362	14,818,338	14,838,357	20,019	0.127	0.118	0.930

The average of percentage of oil desorbed from the surface is 0.935 ± 0.00451.

Table 65: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.100% SDS in low salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	16.8	953.4	936.6
Run2	15.7	102.3	86.6
Run3	15.2	1163.8	1148.6

The average dissipation increase after coating is around 723.9 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

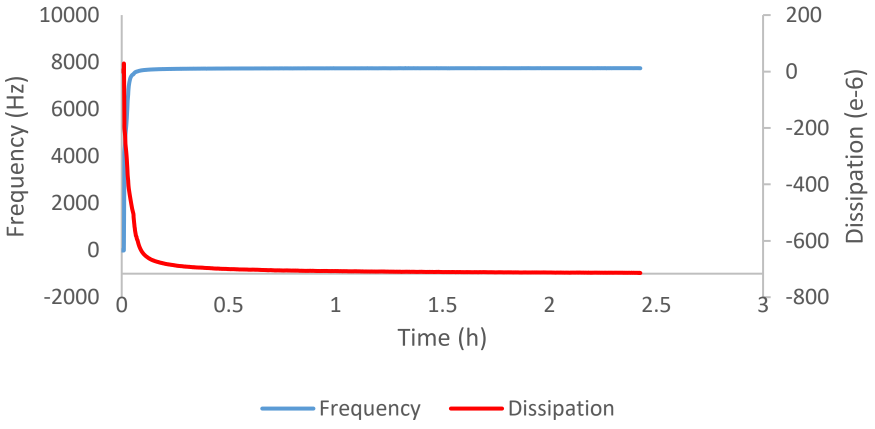


Figure 48: A representative plot showing the change in frequency and dissipation versus time for 0.100% SDS in low salinity.

0.200% SDS

Table 66: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.200 % SDS in low salinity

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	113.538	113.825	0.287
Run2	114.468	114.744	0.276
Run3	115.721	116.027	0.306

For 0.005% SDS in low salinity, the average of oil adsorbed on the surface is 0.290 ± 0.0005 mg.

Table 67: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.200% SDS in low salinity

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,847,728	14,823,121	14,823,057	14,846,323	23,266	0.145	0.137	0.946
2	14,852,696	14,826,317	14,826,312	14,850,683	24,371	0.156	0.144	0.924
3	14,837,840	14,818,464	14,818,005	14,836,323	18,318	0.114	0.108	0.945

The average ratio of oil desorbed from the surface is 0.938 ± 0.0124.

Table 68: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.200% SDS in low salinity

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.0	823.4	808.4
Run2	17.1	184.0	166.9
Run3	15.5	1048.3	1032.8

The average dissipation increase after coating is around 669.4 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

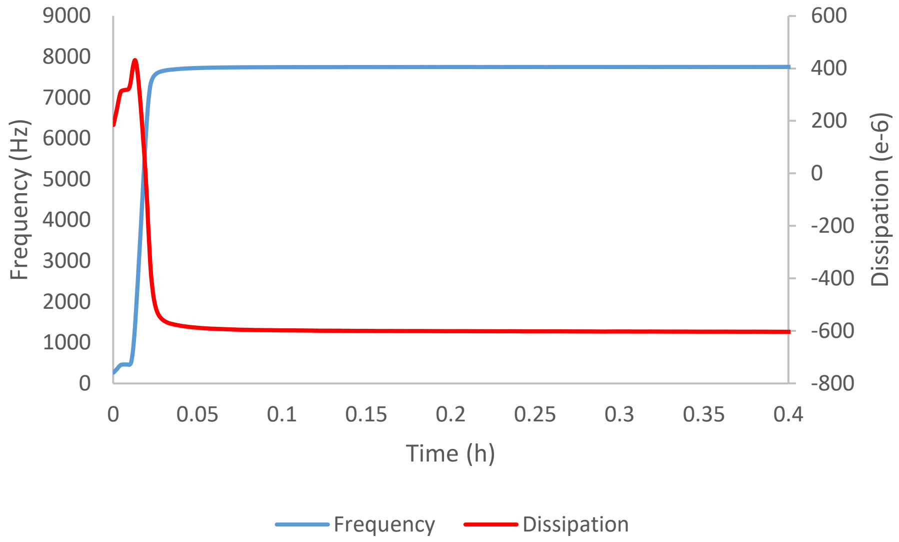


Figure 49: A representative plot showing the change in frequency and dissipation versus time for 0.200% SDS in low salinity

c. <u>0.6% NaCl</u>

0.002% SDS

Table 69: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.002 % SDS in moderate salinity.

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	115.712	116.121	0.409
Run2	114.084	114.496	0.412
Run3	112.946	113.353	0.407

For 0.002% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.409 ± 0.0005 mg.

Table 70: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.002% SDS in moderate salinity

	F $_{clean}$ (Hz)	F $_{coated}$ (Hz)	F $_{before\ desorption}$ (Hz)	F $_{after\ desorption}$ (Hz)	ΔF $_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,838,213	14,819,675	14,819,719	14,835,637	15,918	0.109	0.0939	0.859
2	14,853,795	14,839,103	14,839,149	14,851,697	12,548	0.0867	0.0740	0.854
3	14,845,083	14,823,050	14,823,017	14,841,999	18,982	0.130	0.112	0.862

The average of percentage of oil desorbed from the surface is 0.858 ± 0.00404.

Table 71: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.002% SDS in moderate salinity

	D $_{clean}$ (e-6)	D $_{coated}$ (e-6)	ΔD $_{adsorption}$ (e-6)
Run1	15.2	1007.2	992
Run2	16.3	402.1	385.8
Run3	17.3	989.7	972.4

The average dissipation increase after coating is around 783.4 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

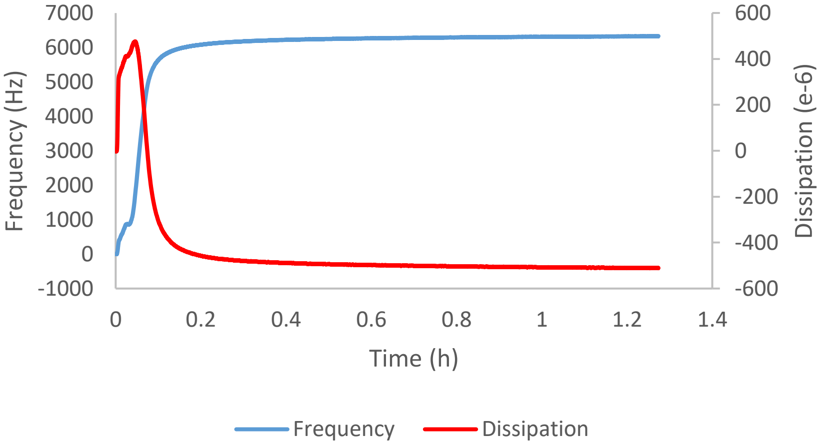


Figure 50: A representative plot showing the change in frequency and dissipation versus time for 0.002% SDS in moderate salinity

0.005% SDS

Table 72: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.005% SDS in moderate salinity.

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.220	116.456	0.236
Run2	115.630	115.968	0.338
Run3	114.809	115.199	0.390

For 0.005% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.321 ± 0.0005 mg.

Table 73: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.005% SDS in moderate salinity

	F clean (Hz)	**F coated (Hz)**	**F before desorption (Hz)**	**F after desorption (Hz)**	**ΔF desorbed (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,841,492	14,825,306	14,824,852	14,839,916	15,064	0.0955	0.0889	0.931
2	14,839,372	14,817,114	14,817,716	14,837,862	20,146	0.131	0.119	0.905
3	14,863,320	14,843,030	14,843,058	14,860,777	17,719	0.120	0.105	0.873

The average of percentage of oil desorbed from the surface is 0.903 ± 0.0291.

Table 74: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.005% SDS in moderate salinity

	D clean (e-6)	**D coated (e-6)**	**ΔD adsorption (e-6)**
Run1	19	885.2	866.2
Run2	18.7	781.8	763.1
Run3	23.1	691.3	668.2

The average dissipation increase after coating is around 765.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

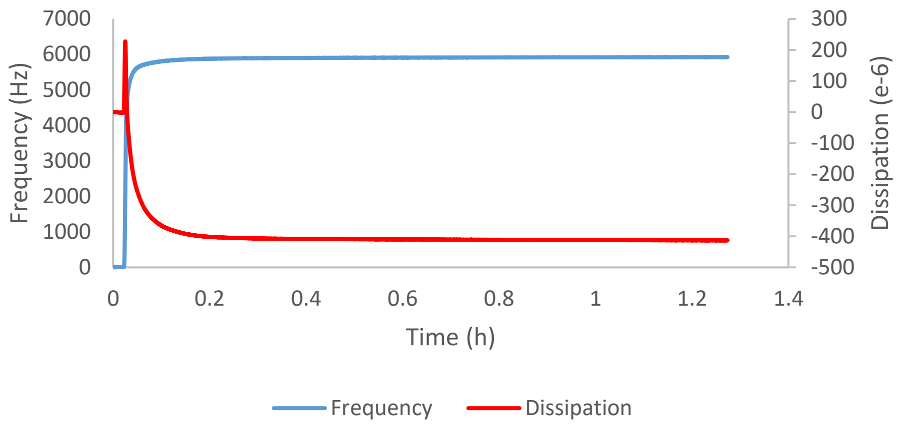


Figure 51: A representative plot showing the change in frequency and dissipation versus time for 0.005% SDS in moderate salinity

0.010% SDS

Table 75: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.010 % SDS in moderate salinity

	W $_{\text{clean}}$ (mg)	**W $_{\text{coated}}$ (mg)**	**Adsorption (mg)**
Run1	116.105	116.366	0.261
Run2	114.748	115.000	0.252
Run3	114.786	115.002	0.216

For 0.010% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.243 ± 0.0005 mg.

Table 76: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.010% SDS in moderate salinity

	F $_{\text{clean}}$ (Hz)	**F $_{\text{coated}}$ (Hz)**	**F $_{\text{before desorption}}$ (Hz)**	**F $_{\text{after desorption}}$ (Hz)**	**ΔF $_{\text{desorbed}}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,842,400	14,820,325	14,820,196	14,840,074	19,878	0.130	0.117	0.900
2	14,863,215	14,842,932	14,842,900	14,861,643	18,743	0.120	0.111	0.924
3	14,839,959	14,825,928	14,825,748	14,838,474	12,726	0.0828	0.0751	0.907

The average ratio of oil desorbed from the surface is 0.911 ± 0.0123.

Table 77: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.010% SDS in moderate salinity

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	15	856	841
Run2	15	497.6	482.6
Run3	15.7	444.3	428.6

The average dissipation increase after coating is around 584.1 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

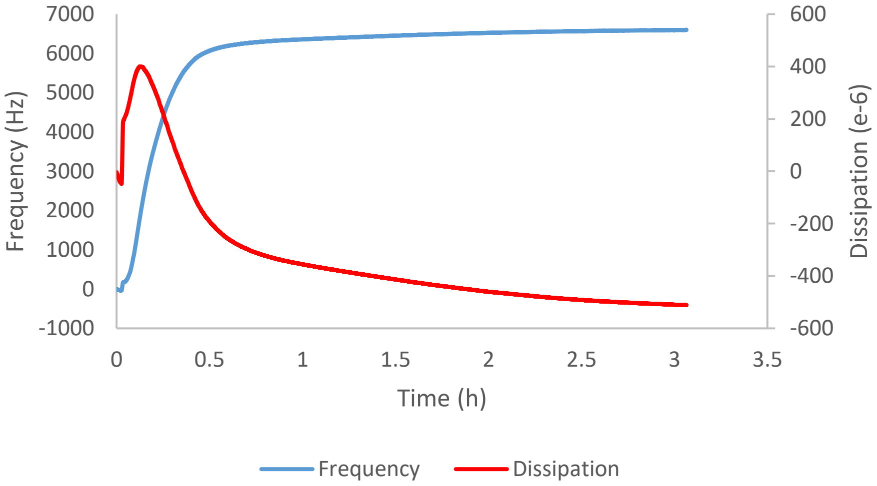


Figure 52: A representative plot showing the change in frequency and dissipation versus time for 0.010% SDS in moderate salinity

0.050% SDS

Table 78: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.050 % SDS in moderate salinity.

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run1	114.810	115.129	0.319
Run2	113.085	113.300	0.215
Run3	114.807	115.140	0.333

For 0.050% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.289 ± 0.0005 mg.

Table 79: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.050% SDS in moderate salinity

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,840,097	14,811,914	14,811,206	14,837,838	26,632	0.166	0.157	0.945
2	14,846,450	14,826,816	14,826,209	14,844,973	18,764	0.116	0.111	0.956
3	14,840,045	14,821,463	14,821,698	14,838,417	16,719	0.110	0.0986	0.900

The average ratio of oil desorbed from the surface is 0.933 ± 0.0297.

Table 80: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.0502% SDS in moderate salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	15.4	680.4	665
Run2	16.8	470.9	454.1
Run3	66	933.5	867.5

The average dissipation increase after coating is around 662.2 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

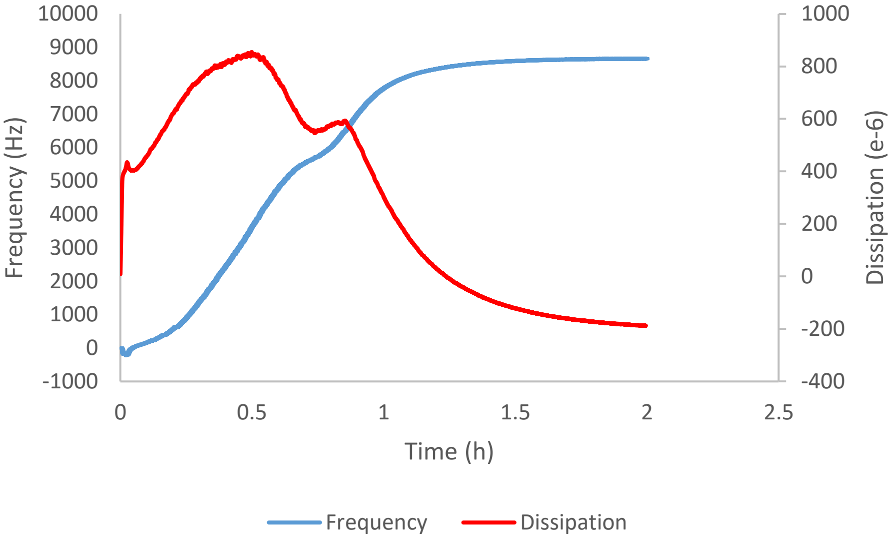


Figure 53: A representative plot showing the change in frequency and dissipation versus time for 0.050% SDS in moderate salinity

0.100% SDS

Table 81: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.100 % SDS in moderate salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	113.097	113.340	0.243
Run2	112.951	113.213	0.262
Run3	114.812	115.240	0.428

For 0.100% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.311 ± 0.0005 mg.

Table 82: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.100% SDS in moderate salinity

	F clean (Hz)	**F coated (Hz)**	**F before desorption (Hz)**	**F after desorption (Hz)**	**ΔF desorbed (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,846,324	14,823,457	14,823,242	14,844,827	21,585	0.135	0.127	0.944
2	14,845,453	14,821,122	14,821,106	14,843,856	22,750	0.144	0.134	0.935
3	14,863,124	14,846,885	14,847,004	14,861,977	14,973	0.0958	0.0883	0.922

The average ratio of oil desorbed from the surface is 0.934 ± 0.0111.

Table 83: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.100% SDS in moderate salinity

	D clean (e-6)	**D coated (e-6)**	**ΔD adsorption (e-6)**
Run1	16.0	760.0	744.0
Run2	15.2	778.8	763.6
Run3	21.0	921.3	900.3

The average dissipation increase after coating is around 802.6 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

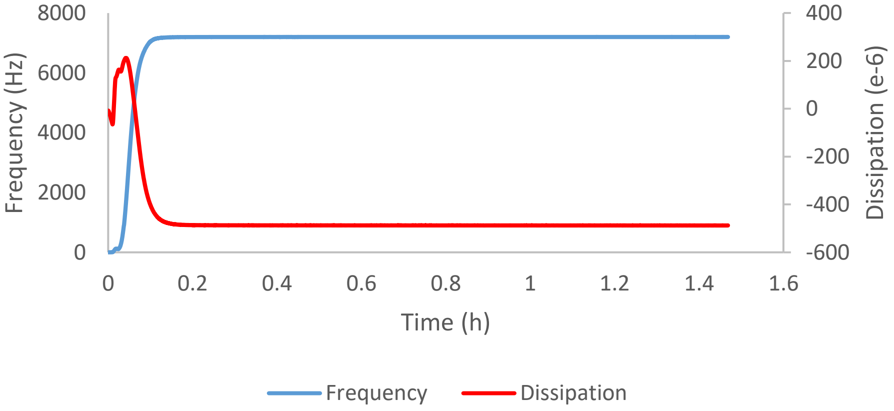


Figure 54: A representative plot showing the change in frequency and dissipation versus time for 0.100% SDS in moderate salinity

0.200% SDS

Table 84: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.200 % SDS in moderate salinity

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	115.627	115.867	0.240
Run2	114.088	114.325	0.237
Run3	112.955	113.183	0.228

For 0.200% SDS in moderate salinity, the average of oil adsorbed on the surface is 0.235 ± 0.0005 mg.

Table 85: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.200% SDS in moderate salinity

	F $_{clean}$ (Hz)	**F $_{coated}$ (Hz)**	**F $_{before\ desorption}$ (Hz)**	**F $_{after\ desorption}$ (Hz)**	**ΔF $_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,839,367	14,818,582	14,818,577	14,837,957	19,380	0.123	0.114	0.932
2	14,854,282	14,834,289	14,834,150	14,852,885	18,735	0.118	0.111	0.937
3	14,845,444	14,829,078	14,828,511	14,844,028	15,517	0.0966	0.0916	0.948

The average ratio of oil desorbed from the surface is 0.939 ± 0.00819.

Table 86: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.200% SDS in moderate salinity

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	16.2	595.8	579.6
Run2	17.1	466.4	449.3
Run3	14.9	245.9	231.0

The average dissipation increase after coating is around 420.0 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

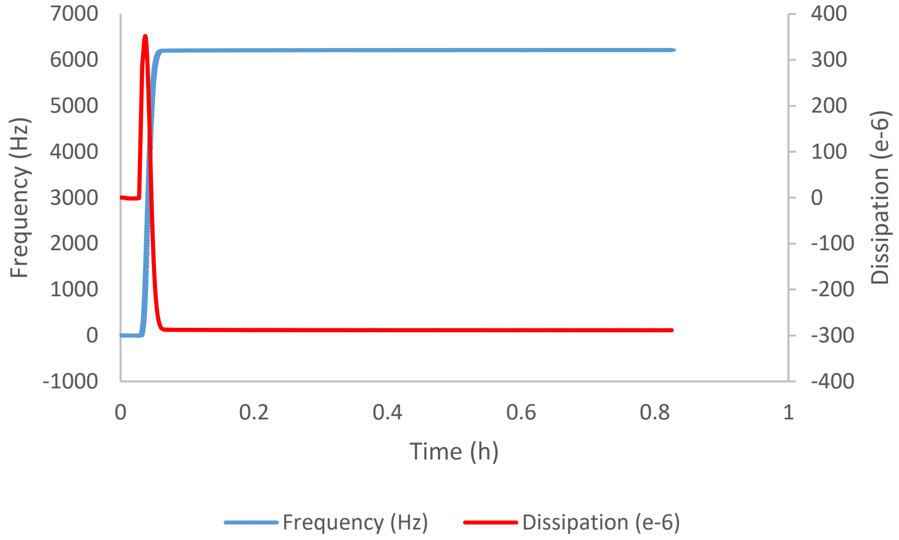


Figure 55: A representative plot showing the change in frequency and dissipation versus time for 0.200% SDS in moderate salinity.

3. Triton X-100

a. <u>0% NaCl</u>

0.002% Triton X-100

Table 87: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.002 % Triton X-100 in distilled water

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.105	116.220	0.115
Run2	114.078	114.354	0.276
Run3	116.214	116.586	0.372

For 0.002% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.254 ± 0.0005 mg.

Table 88: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.002% Triton X-100 in distilled water

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,842,336	14,819,724	14,819,663	14,838,296	18,633	0.133	0.110	0.824
2	14,854,248	14,833,873	14,833,919	14,850,644	16,725	0.120	0.0987	0.821
3	14,842,302	14,821,128	14,821,137	14,839,722	18,585	0.125	0.110	0.878

The average ratio of oil desorbed from the surface is 0.841 ± 0.0321.

Table 89: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.002% Triton X-100 in distilled water

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	15	802.9	787.9
Run2	14.9	849.1	834.2
Run3	15	1011.1	996.1

The average dissipation increase after coating is around 871.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

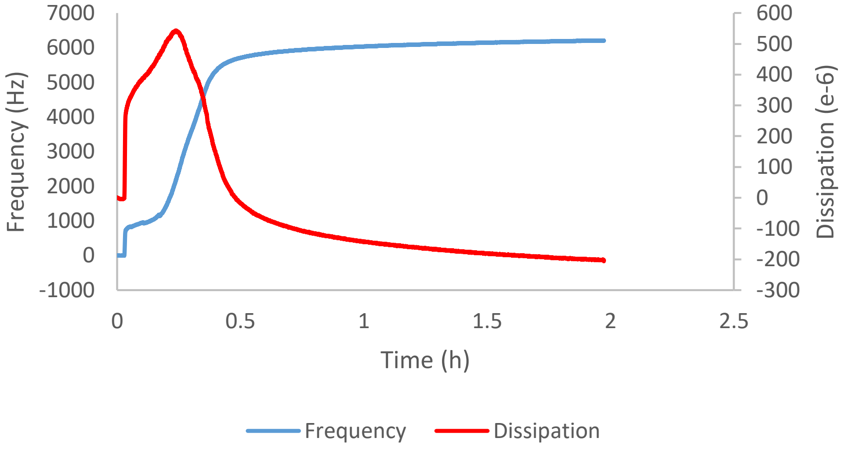


Figure 56: A representative plot showing the change in frequency and dissipation versus time for 0.002% Triton X-100 in distilled water

0.005% Triton X-100

Table 90: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.005 % Triton X-100 in distilled water

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	116.108	116.298	0.19
Run2	114.089	114.256	0.167
Run3	114.084	114.276	0.192

For 0.005% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.183 ± 0.0005 mg.

Table 91: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.002% Triton X-100 in distilled water

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,842,417	14,825,035	14,825,075	14,840,711	15,636	0.103	0.0923	0.900
2	14,854,417	14,841,463	14,840,361	14,850,705	10,344	0.0764	0.0610	0.799
3	14,854,000	14,835,501	14,835,455	14,852,102	16,647	0.109	0.0982	0.900

The average ratio of oil desorbed from the surface is 0.866 ± 0.0583.

Table 92: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.002% Triton X-100 in distilled water

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.1	419.4	404.3
Run2	16.4	182.7	166.3
Run3	22.4	1035.3	1012.9

The average dissipation increase after coating is around 527.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

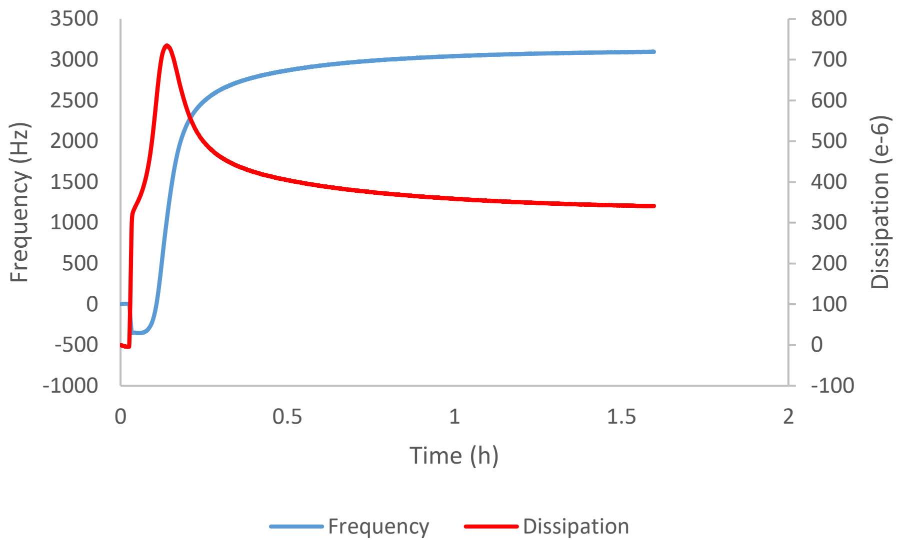


Figure 57: A representative plot showing the change in frequency and dissipation versus time for 0.005% Triton X-100 in distilled water

0.010% Triton X-100

Table 93: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.010 % Triton X-100 in distilled water.

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	112.95	113.215	0.265
Run2	116.208	116.398	0.19
Run3	116.102	116.471	0.369

For 0.010% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.275 ± 0.0005 mg.

Table 94:The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.010% Triton X-100 in distilled water

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,845,269	14,827,159	14,827,147	14,843,482	16,335	0.107	0.0964	0.902
2	14,841,885	14,823,524	14,823,608	14,839,806	16,198	0.108	0.0956	0.882
3	14,842,528	14,820,918	14,820,918	14,839,999	19,081	0.127	0.113	0.883

The average ratio of oil desorbed from the surface is 0.889 ± 0.0113.

Table 95: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.010% Triton X-100 in distilled water

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	16.8	895.1	878.3
Run2	16.1	978.5	962.4
Run3	15.4	1048.9	1033.5

The average dissipation increase after coating is around 958.1 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

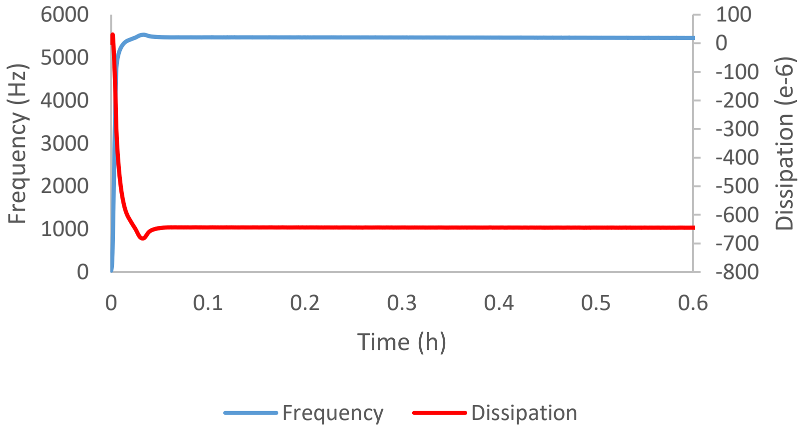


Figure 58: A representative plot showing the change in frequency and dissipation versus time for 0.010% Triton X-100 in distilled water

0.015% Triton X-100

Table 96: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.015 % Triton X-100 in distilled water

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run1	114.876	115.092	0.216
Run2	113.099	113.378	0.279
Run3	115.537	115.755	0.218

For 0.015% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.238 ± 0.0005 mg**.**

Table 97: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.015% Triton X-100 in distilled water

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,403	14,825,063	14,823,226	14,835,066	11,840	0.0846	0.0699	0.826
2	14,846,335	14,829,232	14,828,832	14,843,947	15,115	0.101	0.0892	0.884
3	14,838,493	14,820,688	14,820,564	14,836,671	16,107	0.105	0.0950	0.905

The average ratio of oil desorbed from the surface is 0.871 ± 0.0409.

Table 98: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.015% Triton X-100 in distilled water

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	14.8	932.1	917.3
Run2	15.6	709.9	694.3
Run3	15.9	587.4	571.5

The average dissipation increase after coating is around 727.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs**.**

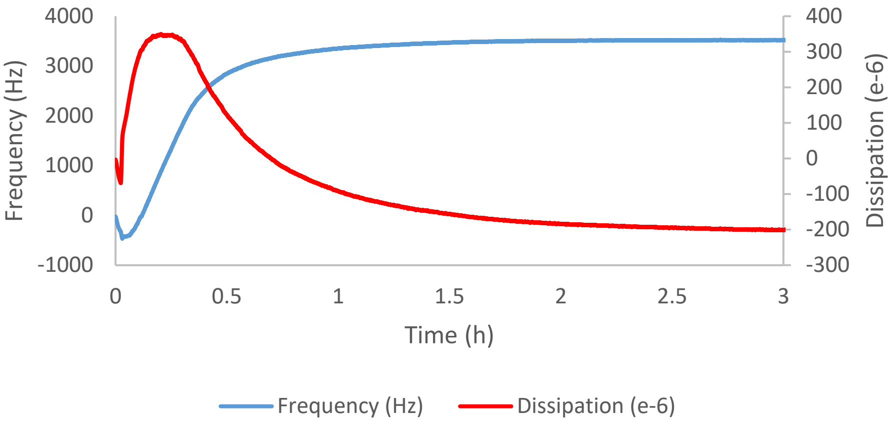


Figure 59: A representative plot showing the change in frequency and dissipation versus time for 0.015% Triton X-100 in distilled water

0.020% Triton X-100

Table 99: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.020 % Triton X-100 in distilled water.

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	113.544	113.807	0.263
Run2	115.720	115.981	0.261
Run3	114.462	114.736	0.274

For 0.020% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.266 ± 0.0005 mg.

Table 100: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.020% Triton X-100 in distilled water

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,848,084	14,825,776	14,825,702	14,845,368	19,666	0.132	0.116	0.882
2	14,838,022	14,821,849	14,821,210	14,835,065	13,855	0.0954	0.0817	0.857
3	14,852,728	14,830,945	14,830,897	14,850,112	19,215	0.129	0.113	0.882

The average ratio of oil desorbed from the surface is 0.873 ± 0.0144.

Table 101: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.020% Triton X-100 in distilled water

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.5	825.9	810.4
Run2	14.9	424.7	409.8
Run3	15.8	648.7	632.9

The average dissipation increase after coating is around 617.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

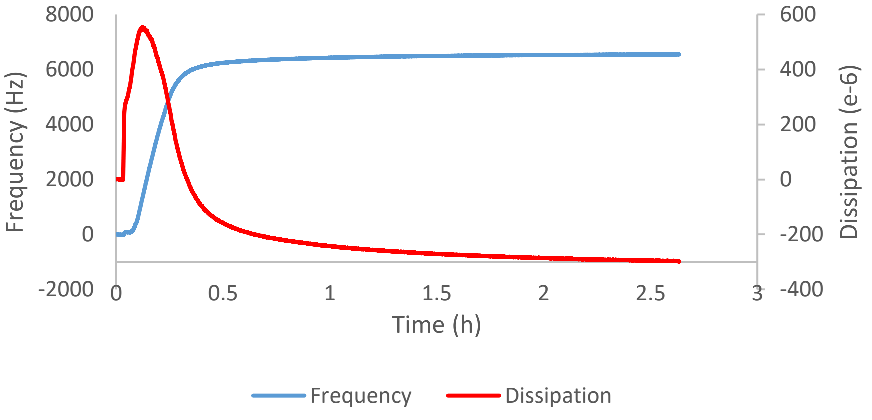


Figure 60: A representative plot showing the change in frequency and dissipation versus time for 0.020% Triton X-100 in distilled water

0.040% Triton X-100

Table 102: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.040 % Triton X-100 in distilled water.

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	116.183	116.395	0.212
Run2	114.876	115.105	0.229
Run3	116.214	116.470	0.256

For 0.040% Triton X-100 in distilled water, the average of oil adsorbed on the surface is 0.232 ± 0.0005 mg.

Table 103: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.040% Triton X-100 in distilled water

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,842,104	14,828,176	14,826,886	14,840,052	13,166	0.0822	0.0777	0.945
2	14,839,249	14,818,045	14,817,987	14,837,303	19,316	0.125	0.114	0.911
3	14,841,980	14,821,685	14,821,547	14,840,671	19,124	0.120	0.113	0.942

The average ratio of oil desorbed from the surface is 0.933 ± 0.0188.

Table 104: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.040% Triton X-100 in distilled water

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.4	164.6	149.2
Run2	16.1	470.2	454.1
Run3	14.8	1158.6	1143.8

The average dissipation increase after coating is around 582.4 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

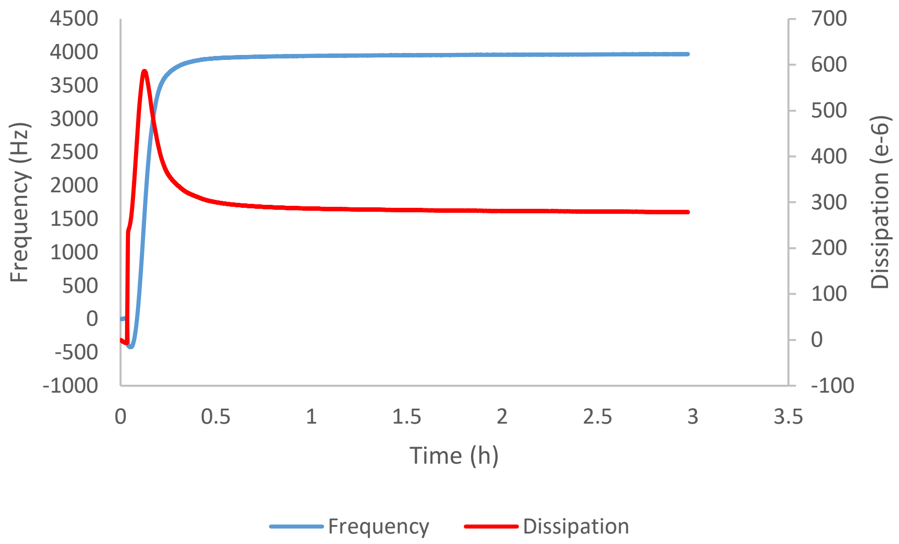


Figure 61: A representative plot showing the change in frequency and dissipation versus time for 0.040% Triton X-100 in distilled water

b. <u>0.2% NaCl</u>

0.001% Triton X-100

Table 105: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.001 % Triton X-100 in low salinity.

	W_{clean} (mg)	**W_{coated} (mg)**	**Adsorption (mg)**
Run1	116.109	116.548	0.439
Run2	114.095	114.307	0.212
Run3	116.108	116.377	0.269

For 0.001% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.307 ± 0.0005 mg.

Table 106: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.001% Triton X-100 in low salinity.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,841,966	14,825,621	14,825,805	14,840,135	14,330	0.0964	0.0845	0.877
2	14,853,733	14,843,666	14,843,988	14,852,575	8,587	0.0594	0.0507	0.853
3	14,842,378	14,823,035	14,822,992	14,840,668	17,676	0.114	0.104	0.914

The average ratio of oil desorbed from the surface is 0.881 ± 0.0307.

Table 107: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.001% Triton X-100 in low salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	14.9	680.2	665.3
Run2	16.8	822.2	805.4
Run3	16.1	676.4	660.3

The average dissipation increase after coating is around 710.3 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

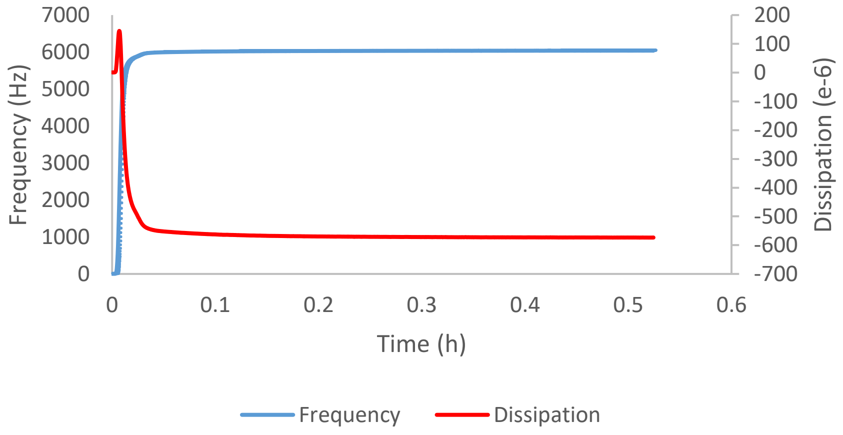


Figure 62: A representative plot showing the change in frequency and dissipation versus time for 0.001% Triton X-100 in low salinity

0.002% Triton X-100

Table 108: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.002 % Triton X-100 in low salinity.

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	115.595	115.989	0.394
Run2	114.743	115.030	0.287
Run3	114.763	115.163	0.400

For 0.002% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.360 ± 0.0005 mg.

Table 109: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.002% Triton X-100 in low salinity.

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,565	14,818,676	14,818,682	14,837,359	18,677	0.123	0.110	0.894
2	14,863,351	14,843,321	14,843,347	14,861,443	18,096	0.118	0.107	0.903
3	14,839,861	14,821,155	14,821,161	14,838,472	17,311	0.110	0.102	0.925

The average ratio of oil desorbed from the surface is 0.908 ± 0.0159.

Table 110: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.002% Triton X-100 in low salinity

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.0	1167.7	1152.7
Run2	15.0	801.4	786.4
Run3	16.4	803.7	787.3

The average dissipation increase after coating is around 908.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

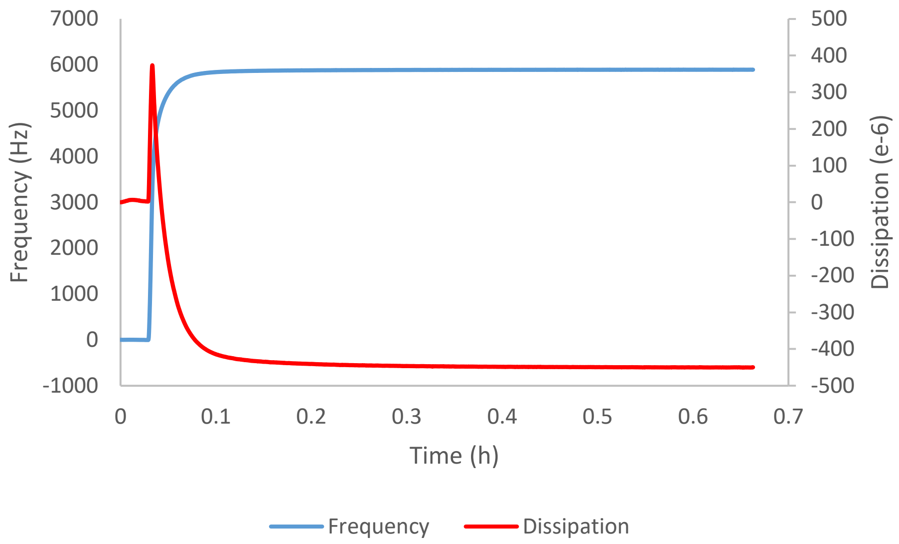


Figure 63: A representative plot showing the change in frequency and dissipation versus time for 0.002% Triton X-100 in low salinity

0.005% Triton X-100

Table 111: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.005 % Triton X-100 in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.209	116.637	0.428
Run2	115.538	115.818	0.280
Run3	114.744	114.962	0.218

For 0.005% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.309 ± 0.0005 mg.

Table 112: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.005% Triton X-100 in low salinity.

	F_{clean} (Hz)	**F_{coated} (Hz)**	**$F_{before\ desorption}$ (Hz)**	**$F_{after\ desorption}$ (Hz)**	**$\Delta F_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,841,535	14,822,689	14,822,672	14,840,593	17,921	0.111	0.106	0.951
2	14,838,003	14,822,957	14,822,859	14,836,361	13,502	0.0888	0.0797	0.897
3	14,863,082	14,847,337	14,847,388	14,861,584	14,196	0.0929	0.0838	0.902

The average ratio of oil desorbed from the surface is 0.917 ± 0.0298.

Table 113: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.005% Triton X-100 in low salinity.

	D_{clean} (e-6)	**D_{coated} (e-6)**	**$\Delta D_{adsorption}$ (e-6)**
Run1	34.4	742.9	708.5
Run2	16.0	546.7	530.7
Run3	17.1	714.9	697.8

The average dissipation increase after coating is around 645.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

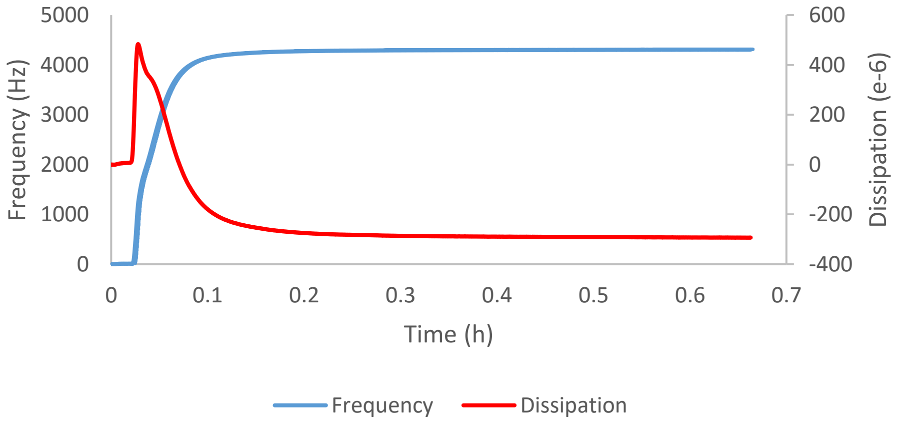


Figure 64: A representative plot showing the change in frequency and dissipation versus time for 0.005% Triton X-100 in low salinity

0.010% Triton X-100

Table 114: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.010 % Triton X-100 in low salinity

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	115.628	115.911	0.283
Run2	116.127	116.365	0.238
Run3	115.626	115.887	0.261

For 0.010% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.261 ± 0.0005 mg.

Table 115: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.010% Triton X-100 in low salinity.

	F $_{clean}$ (Hz)	**F $_{coated}$ (Hz)**	**F $_{before\ desorption}$ (Hz)**	**F $_{after\ desorption}$ (Hz)**	**ΔF $_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,839,405	14,822,563	14,822,534	14,837,905	15,371	0.0994	0.0907	0.913
2	14,842,024	14,822,133	14,822,071	14,840,505	18,434	0.117	0.109	0.927
3	14,839,082	14,819,744	14,819,730	14,837,785	18,055	0.114	0.107	0.934

The average ratio of oil desorbed from the surface is 0.927 ± 0.0107.

Table 116: : The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.010% Triton X-100 in low salinity

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	15.7	646	630.3
Run2	15.1	670.5	655.4
Run3	17.2	639	621.8

The average dissipation increase after coating is around 635.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

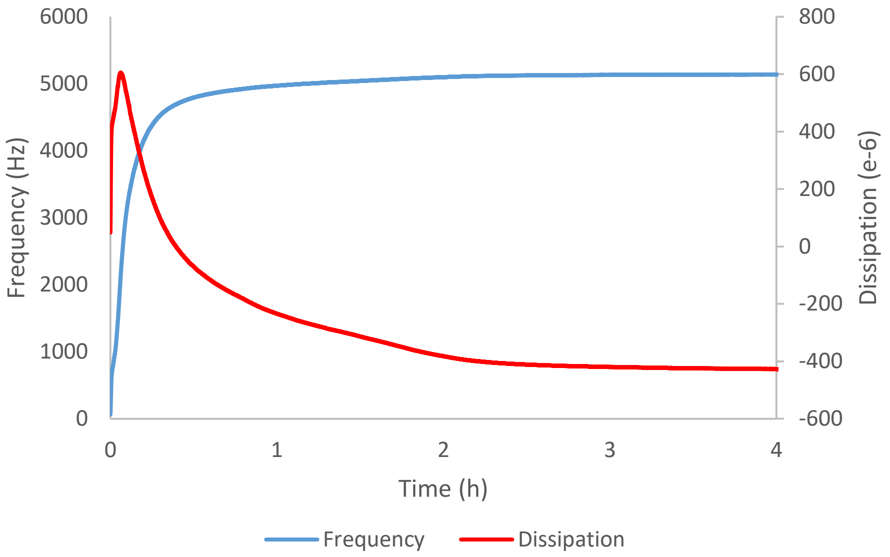


Figure 65: A representative plot showing the change in frequency and dissipation versus time for 0.010% Triton X-100 in low salinity

0.015% Triton X-100

Table 117: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.015 % Triton X-100 in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	114.806	115.083	0.277
Run2	115.627	115.965	0.338
Run3	116.108	116.475	0.367

For 0.015% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.327 ± 0.0005 mg.

Table 118: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.015% Triton X-100 in low salinity.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{\text{before desorption}}$ (Hz)	$F_{\text{after desorption}}$ (Hz)	$\Delta F_{\text{desorbed}}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,494	14,820,296	14,820,290	14,838,068	17,778	0.113	0.105	0.926
2	14,839,450	14,816,048	14,816,035	14,837,427	21,392	0.138	0.126	0.914
3	14,841,981	14,820,062	14,820,124	14,840,720	20,596	0.129	0.122	0.940

The average ratio of oil desorbed from the surface is 0.927 ± 0.0130.

Table 119: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.015% Triton X-100 in low salinity.

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{\text{adsorption}}$ (e-6)
Run1	29.4	478.4	449.0
Run2	15.5	1051.6	1036.1
Run3	15.1	821.5	806.4

The average dissipation increase after coating is around 763.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

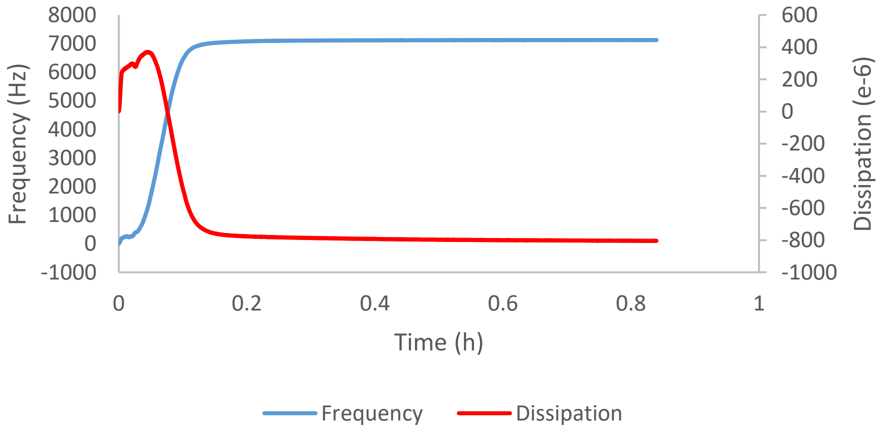


Figure 66: A representative plot showing the change in frequency and dissipation versus time for 0.015% Triton X-100 in low salinity

0.020% Triton X-100

Table 120: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.020 % Triton X-100 in low salinity

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	114.817	115.041	0.224
Run2	114.810	115.038	0.228
Run3	114.773	115.055	0.282

For 0.020% Triton X-100 in low salinity, the average of oil adsorbed on the surface is 0.245 ± 0.0005 mg.

Table 121: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.020% Triton X-100 in low salinity.

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,863,218	14,842,604	14,842,507	14,861,326	18,819	0.122	0.111	0.913
2	14,863,094	14,841,381	14,841,137	14,861,579	20,442	0.128	0.121	0.941
3	14,839,790	14,819,783	14,819,757	14,838,422	18,665	0.118	0.110	0.933

The average ratio of oil desorbed from the surface is 0.929 ± 0.0144.

Table 122: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.020% Triton X-100 in low salinity

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.4	776.9	761.5
Run2	16.5	464.7	448.2
Run3	17.2	791.3	774.1

The average dissipation increase after coating is around 661.3 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

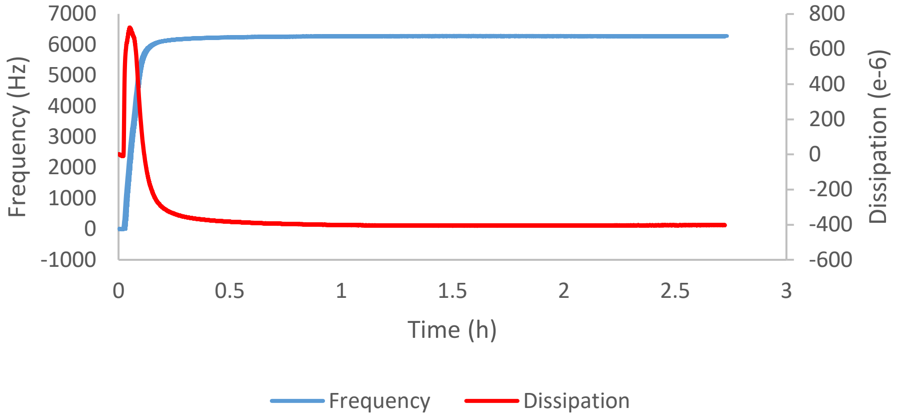


Figure 67: A representative plot showing the change in frequency and dissipation versus time for 0.020% Triton X-100 in low salinity

c. 0.6% NaCl

0.001% Triton X-100

Table 123: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.001 % Triton X-100 in moderate salinity.

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run1	116.112	116.302	0.190
Run2	114.092	114.293	0.201
Run3	114.082	114.333	0.251

For 0.001% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.214 ± 0.0005 mg.

Table 124: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.001% Triton X-100 in moderate salinity.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,842,336	14,828,039	14,827,953	14,840,473	12,520	0.0844	0.0739	0.876
2	14,854,171	14,837,556	14,837,400	14,852,483	15,083	0.0980	0.0890	0.908
3	14,854,220	14,836,580	14,836,321	14,852,043	15,722	0.104	0.0928	0.891

The average ratio of oil desorbed from the surface is 0.892 ± 0.0160.

Table 125: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.001% Triton X-100 in moderate salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	15.3	495.1	479.8
Run2	15.1	693.1	678.0
Run3	15.3	851.2	835.9

The average dissipation increase after coating is around 664.6 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

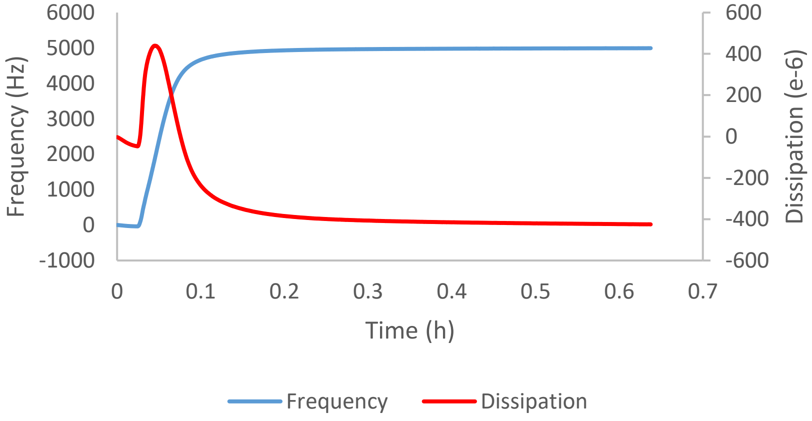


Figure 68: A representative plot showing the change in frequency and dissipation versus time for 0.001% Triton X-100 in moderate salinity

0.0015% Triton X-100

Table 126: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.0015 % Triton X-100 in moderate salinity.

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run1	115.624	115.838	0.214
Run2	112.954	113.195	0.241
Run3	112.942	113.243	0.301

For 0.0015% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.252 ± 0.0005 mg.

Table 127: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.0015% Triton X-100 in moderate salinity

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,523	14,821,238	14,821,233	14,837,647	16,414	0.108	0.0968	0.898
2	14,844,987	14,826,740	14,826,693	14,843,589	16,896	0.108	0.100	0.926
3	14,845,469	14,824,521	14,824,516	14,843,104	18,588	0.124	0.110	0.887

The average ratio of oil desorbed from the surface is 0.904 ± 0.0201.

Table 128: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.0015% Triton X-100 in moderate salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	15.2	1043.7	1028.5
Run2	14.9	878.9	864.0
Run3	14.8	794.5	779.7

The average dissipation increase after coating is around 890.7 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

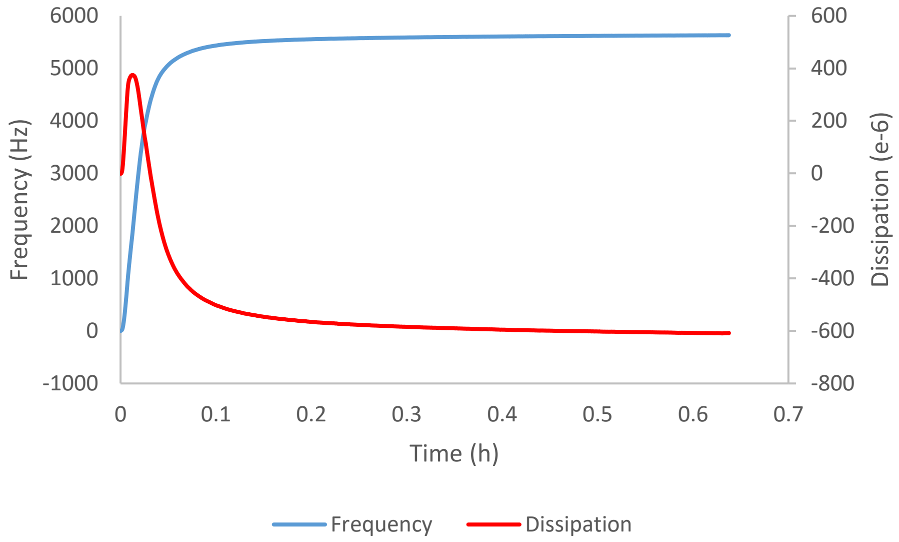


Figure 69: A representative plot showing the change in frequency and dissipation versus time for 0.0015% Triton X-100 in moderate salinity

0.002% Triton X-100

Table 129: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.002 % Triton X-100 in moderate salinity.

	W $_{\text{clean}}$ (mg)	**W $_{\text{coated}}$ (mg)**	**Adsorption (mg)**
Run1	115.712	115.969	0.257
Run2	114.741	114.980	0.239
Run3	116.107	116.434	0.327

For 0.002% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.274 ± 0.0005 mg.

Table 130: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.002% Triton X-100 in moderate salinity.

	F_{clean} (Hz)	**F_{coated} (Hz)**	**$F_{\text{before desorption}}$ (Hz)**	**$F_{\text{after desorption}}$ (Hz)**	**$\Delta F_{\text{desorbed}}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,838,027	14,816,269	14,816,308	14,836,312	20,004	0.128	0.118	0.919
2	14,863,108	14,845,939	14,845,920	14,861,283	15,363	0.101	0.0906	0.895
3	14,842,423	14,822,922	14,822,830	14,840,573	17,743	0.115	0.105	0.910

The average ratio of oil desorbed from the surface is 0.908 ± 0.0121.

Table 131: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.002% Triton X-100 in moderate salinity

	D_{clean} (e-6)	**D_{coated} (e-6)**	**$\Delta D_{\text{adsorption}}$ (e-6)**
Run1	14.8	1081.4	1066.6
Run2	15.4	906.4	891.0
Run3	15.1	1086.5	1071.4

The average dissipation increase after coating is around 1009.67 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

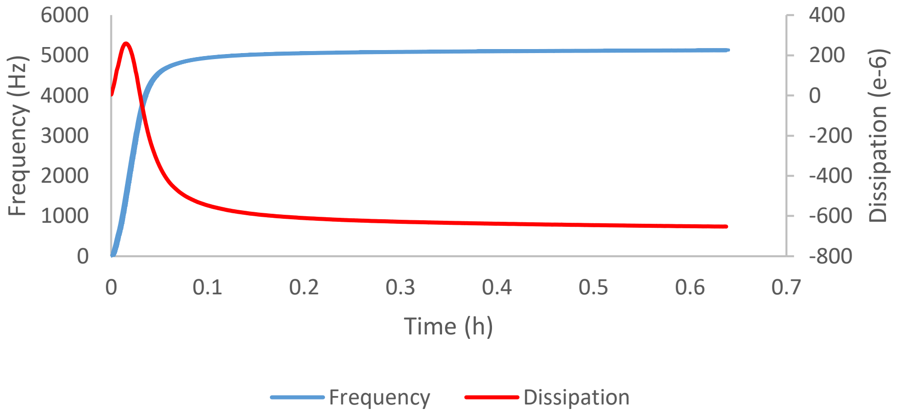


Figure 70: A representative plot showing the change in frequency and dissipation versus time for 0.002% Triton X-100 in moderate salinity

0.005% Triton X-100

Table 132: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.005 % Triton X-100 in moderate salinity.

	W_{clean} (mg)	W_{coated} (mg)	Adsorption (mg)
Run1	115.626	115.960	0.334
Run2	112.945	113.288	0.343
Run3	112.945	113.276	0.331

For 0.005% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.336 ± 0.0005 mg.

Table 133: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.005% Triton X-100 in moderate salinity.

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{before\ desorption}$ (Hz)	$F_{after\ desorption}$ (Hz)	$\Delta F_{desorbed}$ (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,573	14,816,726	14,816,745	14,837,078	20,333	0.135	0.120	0.890
2	14,845,389	14,824,365	14,824,395	14,843,859	19,464	0.124	0.115	0.926
3	14,845,269	14,826,154	14,826,154	14,843,604	17,450	0.113	0.103	0.913

The average ratio of oil desorbed from the surface is 0.910 ± 0.0182.

Table 134: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.005% Triton X-100 in moderate salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$\Delta D_{adsorption}$ (e-6)
Run1	15.4	953.3	937.9
Run2	15.2	977.8	962.6
Run3	15.6	1093.5	1077.9

The average dissipation increase after coating is around 992.8 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

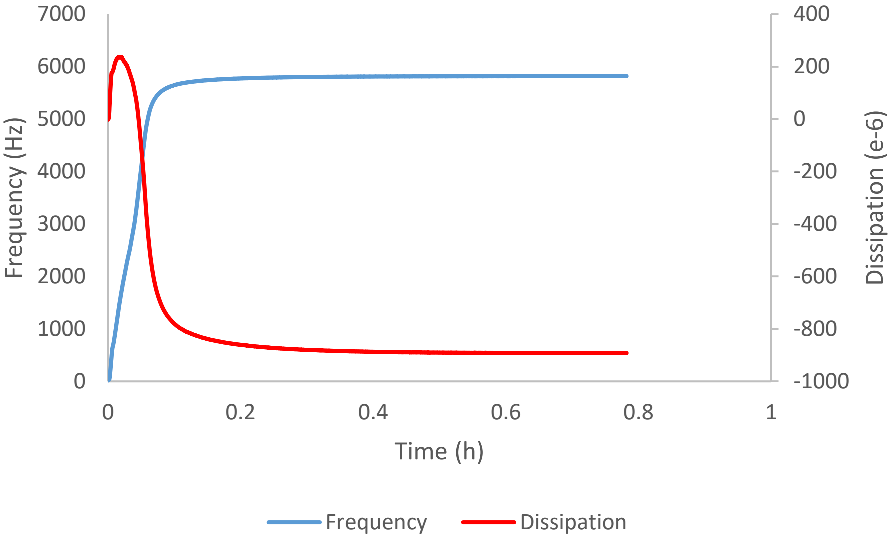


Figure 71: A representative plot showing the change in frequency and dissipation versus time for 0.005% Triton X-100 in moderate salinity

0.010% Triton X-100

Table 135: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.010 % Triton X-100 in moderate salinity.

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run1	114.780	115.164	0.384
Run2	114.050	114.406	0.356
Run3	115.596	115.876	0.280

For 0.010% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.340 ± 0.0005 mg.

Table 136: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.010% Triton X-100 in moderate salinity.

	F $_{clean}$ (Hz)	**F $_{coated}$ (Hz)**	**F $_{before\ desorption}$ (Hz)**	**F $_{after\ desorption}$ (Hz)**	**ΔF $_{desorbed}$ (Hz)**	**Adsorption (ng/cm^2)**	**Desorption (ng/cm^2)**	**% of mass desorbed**
1	14,839,933	14,815,965	14,815,998	14,836,838	20,840	0.141	0.123	0.869
2	14,853,815	14,835,034	14,835,040	14,852,688	17,648	0.111	0.104	0.940
3	14,839,679	14,820,844	14,820,840	14,838,218	17,378	0.111	0.103	0.923

The average ratio of oil desorbed from the surface is 0.911 ± 0.0371.

Table 137: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.010% Triton X-100 in moderate salinity

	D $_{clean}$ (e-6)	**D $_{coated}$ (e-6)**	**ΔD $_{adsorption}$ (e-6)**
Run1	18.9	1028.1	1009.2
Run2	17.0	804.7	787.7
Run3	15.3	990.9	975.6

The average dissipation increase after coating is around 924.2 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

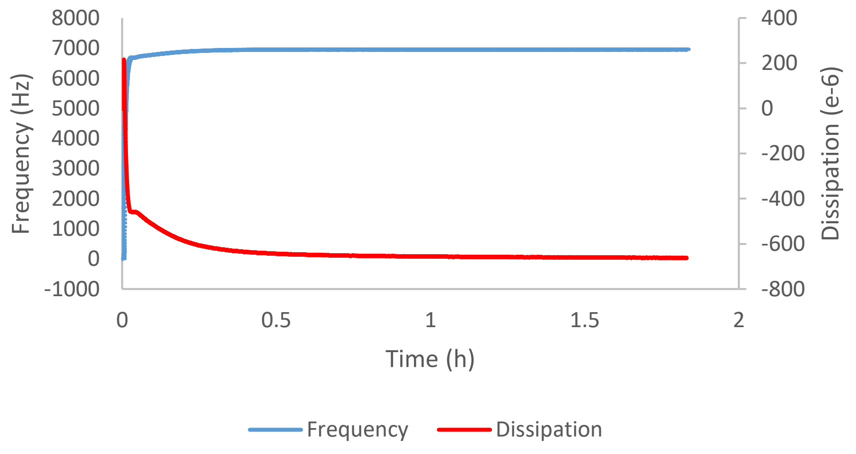


Figure 72: A representative plot showing the change in frequency and dissipation versus time for 0.010% Triton X-100 in moderate salinity

0.015% Triton X-100

Table 138: Weight of silica sensors before and after coating with crude oil B solution for the three runs and the amount of oil adsorbed used for 0.015 % Triton X-100 in moderate salinity.

	W clean (mg)	W coated (mg)	Adsorption (mg)
Run1	114.773	114.972	0.199
Run2	116.206	116.404	0.198
Run3	114.734	115.001	0.267

For 0.015% Triton X-100 in moderate salinity, the average of oil adsorbed on the surface is 0.211 ± 0.0005 mg.

Table 139: The frequency before and after coating, before and after desorption, the amount of oil adsorbed and desorbed with the percentage of mass desorbed from the silica sensor for the three runs used for 0.015% Triton X-100 in moderate salinity.

	F clean (Hz)	F coated (Hz)	F before desorption (Hz)	F after desorption (Hz)	ΔF desorbed (Hz)	Adsorption (ng/cm^2)	Desorption (ng/cm^2)	% of mass desorbed
1	14,839,925	14,823,118	14,823,130	14,838,675	15,545	0.099	0.092	0.925
2	14,841,914	14,831,879	14,831,528	14,840,621	9,093	0.059	0.054	0.906
3	14,863,132	14,840,393	14,840,389	14,861,481	21,092	0.134	0.124	0.928

The average ratio of oil desorbed from the surface is 0.920 ± 0.0120.

Table 140: The dissipation values before and after coating of the silica sensor with the crude oil B solution used for 0.015% Triton X-100 in moderate salinity

	D clean (e-6)	D coated (e-6)	ΔD adsorption (e-6)
Run1	15.3	697.2	681.9
Run2	15.2	181.6	166.4
Run3	20.6	1053.6	1033.0

The average dissipation increase after coating is around 627.1 ± 0.05 e-6.

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

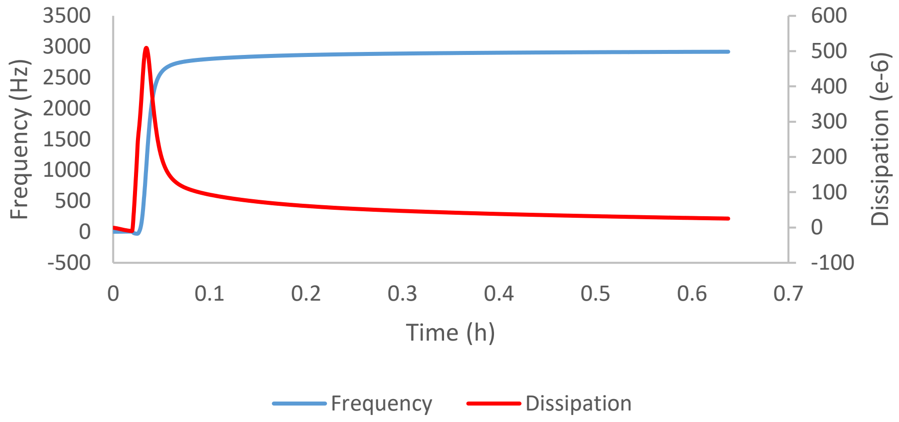


Figure 73: A representative plot showing the change in frequency and dissipation versus time for 0.015% Triton X-100 in moderate salinity

C. Crude oil A

For the crude oil A, only SDS was used as a surfactant for oil removal in distilled water and low salinity solution.

The different SDS concentrations in a certain salinity were performed simultaneously in an increasing order to determine the total amount of crude oil A desorbed from the silica surface.

The temperature remained constant at 20ºC during the runs with a flow rate of 100 µL/min.

For each run in a specific salinity, the weight of the sensor before and after coating was determined in order to see how much oil has adsorbed on the surface in mg.

In addition, the initial frequency, the frequency after coating, the frequency before and after desorption were computed in order to check the amount of oil adsorbed and desorbed from the silica surface in mg/cm^2 using Sauebrey's equation. In this way, the percentage of oil desorbed by SDS and by distilled water can be determined.

The amount of oil desorbed by each SDS concentration, whether in distilled water or in low salinity, was calculated by determining the frequency before and after the injection of that specific concentration

The dissipation before and after coating of the silica sensor with the crude oil A was also computed. The increase in the dissipation indicate the development of a soft molecular film.

Each surfactant concentration in a certain salinity was run separately in order to determine how much this specific amount has desorbed oil on the surface.

1. 0% NaCl

Table 141: Weight of silica sensors before and after coating with crude oil A and the amount of oil adsorbed on the surface for the run in distilled water

	W $_{clean}$ (mg)	**W $_{coated}$ (mg)**	**Adsorption (mg)**
Run 1	116.076	116.639	0.563
Run 2	115.803	116.688	0.885
Run 3	115.951	116.575	0.624

For the first set of experiments where distilled water is the control, an average of 0.691 ± 0.0005 mg of oil was adsorbed to the silica surface.

Table 142: The frequency before, after coating with crude oil A and after the run, the total amount of oil adsorbed and desorbed and the amount desorbed by SDS and distilled water

	F clean (Hz)	F coated (Hz)	F after run (Hz)	ΔF desorbed(Hz)	Adsorption (mg/cm^2)	Total Desorption (mg/cm^2)	Desorbed by SDS (mg/cm^2)	Desorbed by DW (mg/cm^2)
1	14,840,896	14,836,117	14,839,255	3,138	0.0282	0.0185	0.00230	0.0162
2	14,846,460	14,841,786	14,844,719	2,933	0.0276	0.0173	0.00262	0.0147
3	14,860,950	14,855,596	14,860,582	4,986	0.0316	0.0294	0.000596	0.0288

From the frequency obtained, an average of 0.0291 ± 0.00216 mg/cm^2 of oil was adsorbed to the silica surface. Out of 0.0217 ± 0.00667 mg/cm^2 of oil desorbed, an average of 0.00184 ± 0.00109 mg/cm^2 of oil was desorbed using SDS and 0.0199 ± 0.00774 mg/cm^2 desorbed by distilled water.

Table 143: The dissipation values before and after coating of the silica sensor with the crude oil A and after the run with distilled water

	D clean (e-6)	D coated (e-6)	D after run (e-6)	ΔD adsorption (e-6)	ΔD desorption (e-6)
Run 1	16.9	583.8	90.6	566.9	493.2
Run 2	19.8	656.3	87.5	636.5	568.8
Run 3	15.4	452.8	19.2	437.4	433.6

The average dissipation increase after coating is around 546.9 ± 0.05 e-6, while the average dissipation decrease after the run is around 498.5 ± 0.05 e-6.

The amount of oil desorbed by each of the SDS concentrations in distilled water is presented in the tables below.

Run 1

Table 144: Mass of oil desorbed by different SDS concentrations in distilled water in mg/cm^{2}and percentage and the cumulative mass desorbed for run 1.

SDS concentration	**F $_{before}$ (Hz)**	**F $_{after}$ (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,839,760	14,839,787	0.000159	7	0.000159
0.10%	14,839,787	14,839,806	0.000112	5	0.000271
0.20%	14,839,806	14,840,104	0.00176	76	0.00203
0.50%	14,840,104	14,840,137	0.000195	8	0.00222
1.00%	14,840,137	14,840,144	0.0000413	2	0.00227
1.50%	14,840,144	14,840,150	0.0000354	2	0.00230

Run 2

Table 145: Mass of oil desorbed by different SDS concentrations in distilled water in mg/cm2and percentage and the cumulative mass desorbed for run 2.

SDS concentration	**F $_{before}$ (Hz)**	**F $_{after}$ (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,844,728	14,844,765	0.000218	8	0.000218
0.10%	14,844,765	14,844,784	0.000112	4	0.000330
0.20%	14,844,784	14,845,109	0.00192	73	0.00225
0.50%	14,845,109	14,845,157	0.000283	11	0.00253
1.00%	14,845,157	14,845,165	0.0000472	2	0.00258
1.50%	14,845,165	14,845,172	0.0000413	2	0.00262

Run 3

Table 146: Mass of oil desorbed by different SDS concentrations in distilled water in mg/cm2and percentage and the cumulative mass desorbed for run 3.

SDS concentration	**F $_{before}$ (Hz)**	**F $_{after}$ (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,859,288	14,859,290	0.0000118	2	0.0000118
0.10%	14,859,290	14,859,293	0.0000177	3	0.0000295
0.20%	14,859,293	14,859,356	0.0003717	63	0.000401
0.50%	14,859,356	14,859,372	0.0000944	16	0.000496
1.00%	14,859,372	14,859,378	0.0000354	6	0.000531
1.50%	14,859,378	14,859,388	0.0000590	10	0.000592
2.00%	14,859,388	14,859,389	0.0000059	1	0.000596

The frequency and dissipation versus the time is shown in the figure below. This graph will illustrate one of the runs.

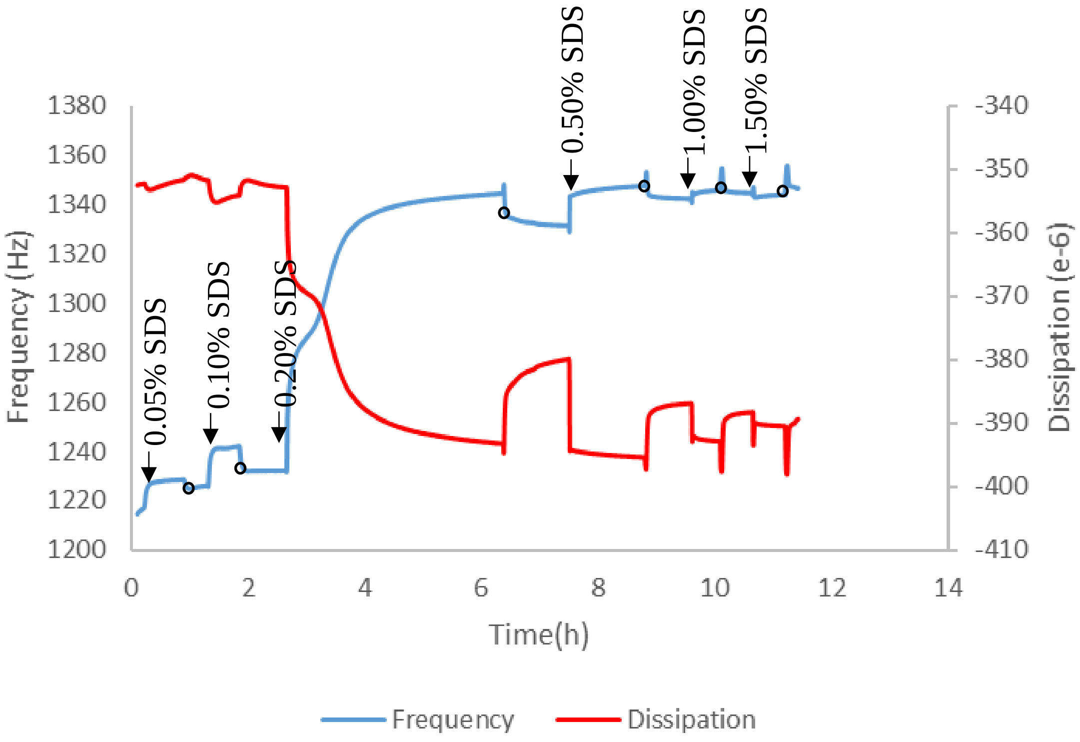


Figure 74: A representative plot showing the change in frequency and dissipation versus time for after injecting different concentrations of surfactants. The black circles represent the injection of distilled water.

2. *0.2% NaCl*

Table 147: Weight of silica sensors before and after coating with crude oil A and the amount of oil adsorbed on the surface for the run in low salinity

	W clean (mg)	**W coated (mg)**	**Adsorption (mg)**
Run 1	116.982	117.803	0.821
Run 2	114.455	115.183	0.728
Run 3	116.077	116.749	0.672

For the second set of experiments where low salinity solution is the control, an average of 0.740 ± 0.0005 mg of oil was adsorbed to the silica surface.

Table 148: The frequency before, after coating with crude oil A and after the run, the total amount of oil adsorbed and desorbed and the amount desorbed by SDS and low salinity

	F_{clean} (Hz)	F_{coated} (Hz)	$F_{after\ run}$ (Hz)	Adsorption (mg/cm^2)	Total Desorption (mg/cm^2)	Desorbed by SDS (mg/cm^2)	Desorbed by LS (mg/cm^2)
1	14,828,397	14,823,570	14,826,164	0.0285	0.0153	0.00172	0.0136
2	14,851,962	14,847,894	14,851,241	0.0240	0.0197	0.000826	0.0189
3	14,840,896	14,836,570	14,838,867	0.0255	0.0136	0.000679	0.0129

From the frequency obtained, an average of 0.0260 ± 0.00229 mg/cm^2 of oil was adsorbed to the silica surface. Out of 0.0162 ± 0.00315 mg/cm^2 of oil desorbed, an average of 0.00110 ± 0.000563 mg/cm^2 of oil was desorbed using SDS and 0.0151 ± 0.00328 mg/cm^2 desorbed by distilled water.

Table 149: The dissipation values before and after coating of the silica sensor with the crude oil A and after the run with low salinity

	D_{clean} (e-6)	D_{coated} (e-6)	$D_{after\ run}$ (e-6)	$\Delta D_{adsorption}$ (e-6)	$\Delta D_{desorption}$ (e-6)
Run 1	16.7	664.4	255.7	647.7	408.7
Run 2	21.5	674.3	223.8	652.8	450.5
Run 3	16.9	462.2	83.6	445.3	378.6

The average dissipation increase after coating is around 581.9 ± 0.05 e-6, while the average dissipation decrease after the run is around 412.6 ± 0.05 e-6.

The amount of oil desorbed by each of the SDS concentrations in distilled water is presented in the tables below.

Run 1

Table 150: Mass of oil desorbed by different SDS concentrations in low salinity in mg/cm2 and percentage and the cumulative mass desorbed for run 1.

SDS concentration	**F_{before} (Hz)**	**F_{after} (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,826,166	14,826,187	0.000124	7.2	0.000124
0.10%	14,826,187	14,826,405	0.00129	74.7	0.00141
0.20%	14,826,405	14,826,434	0.000171	9.9	0.00158
0.50%	14,826,434	14,826,449	0.0000885	5.1	0.00167
1.00%	14,826,449	14,826,456	0.0000413	2.4	0.00171
1.50%	14,826,456	14,826,458	0.0000118	0.7	0.00172

Run 2

Table 151: Mass of oil desorbed by different SDS concentrations in low salinity in mg/cm2 and percentage and the cumulative mass desorbed for run 2.

SDS concentration	**F_{before} (Hz)**	**F_{after} (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,851,242	14,851,248	0.0000354	4.3	0.0000354
0.10%	14,851,248	14,851,328	0.000472	57.1	0.000507
0.20%	14,851,328	14,851,347	0.000112	13.6	0.000619
0.50%	14,851,347	14,851,367	0.000118	14.3	0.000737
1.00%	14,851,367	14,851,375	0.0000472	5.7	0.000785
1.50%	14,851,375	14,851,382	0.0000413	5.0	0.000826

Run 3

Table 152: Mass of oil desorbed by different SDS concentrations in low salinity in mg/cm2 and percentage and the cumulative mass desorbed for run 3.

SDS concentration	**F_{before} (Hz)**	**F_{after} (Hz)**	**Mass desorbed (mg/cm^2)**	**Mass desorbed (by %)**	**Cumulative mass desorbed (mg/cm^2)**
0.05%	14,838,596	14,838,609	0.0000767	11.3	0.0000767
0.10%	14,838,609	14,838,679	0.000413	60.9	0.0004897
0.20%	14,838,679	14,838,689	0.000059	8.7	0.0005487
0.50%	14,838,689	14,838,698	0.0000531	7.8	0.0006018
1.00%	14,838,698	14,838,706	0.0000472	7.0	0.000649
1.50%	14,838,706	14,838,711	0.0000295	4.3	0.0006785

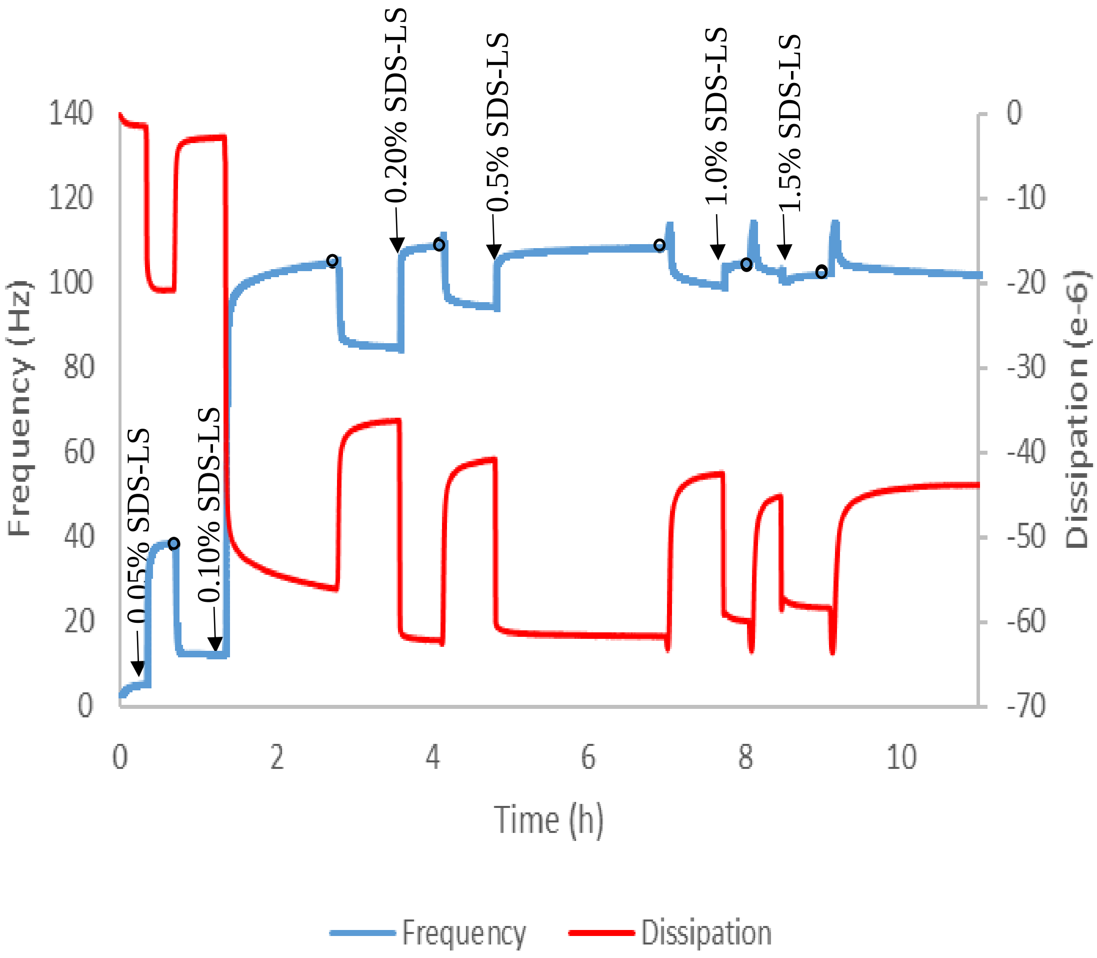


Figure 75: A representative plot showing the change in frequency and dissipation versus time for after injecting different concentrations of surfactants. The black circles represent the injection of low salinity.

CHAPTER IV

DISCUSSION

A. Effect of surfactant concentration

1. Crude oil B

For each run, one concentration of surfactant was injected into the QCM-D without prior washing with the control solution, leading to obtaining the total amount of crude oil B desorbed. This total amount is the amount of oil removed by the control solution and surfactant combined. However, our purpose was to determine how much each concentration of surfactant could contribute to the removal of the oil from the silica surface. For each surfactant, three different sets of experiments were analyzed: with and without salinity. In order to take the washing effect into consideration, the average amount of crude oil B adsorbed and desorbed from the surface were calculated from the results tabulated in the section above. The average amount of crude oil B desorbed by the control solution was obtained by multiplying the average of crude oil adsorbed by the percentage of removal by that control solution itself. To obtain the amount removed by the surfactant, the average amount of oil desorbed was calculated by multiplying the average of oil adsorbed by the change in percentage between the control solution and the total solution containing the surfactant. All these results are summarized in tables along with two graphs: one histogram showing the percentage of oil removed by each of control solution and surfactant concentrations and a general curve showing the total amount desorbed by the surfactant concentrations without washing.

The standard error of the mean method was used to determine the error bars. In addition, ANOVA test was performed in order to check the significance of the error, followed by a Post-hoc Tukey test to check for individual significance.

SDS - DW

Table 153: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by distilled water and different SDS concentrations along with the appropriate percentages

SDS concentration (%)	average amount of crude oil B adsorbed (mg/cm^2)	Average amount of crude oil B desorbed (mg/cm^2)	average amount of crude oil B desorbed by DW (mg/cm^2)	average amount of crude oil B desorbed by SDS (mg/cm^2)	% of crude oil B removed by SDS	Total % of mass desorbed
0	0.0937	0.0618	0.0619	0.0000	0.000	0.663
0.05	0.0832	0.0554	0.0551	0.00000212	0.000025	0.663
0.10	0.0921	0.0807	0.0611	0.0196	0.213	0.876
0.20	0.131	0.123	0.0869	0.0360	0.274	0.937
0.50	0.132	0.124	0.0872	0.0372	0.282	0.946
1.00	0.109	0.102	0.0726	0.0295	0.271	0.932
2.00	0.125	0.116	0.0826	0.0329	0.263	0.927

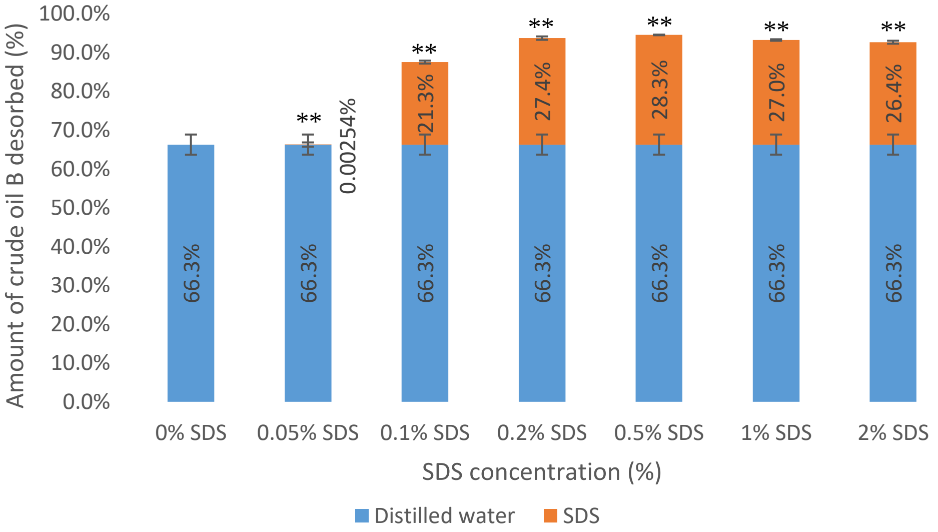


Figure 76: A histogram showing the percentage of crude oil B desorbed from the silica surface by distilled water and different SDS concentrations.

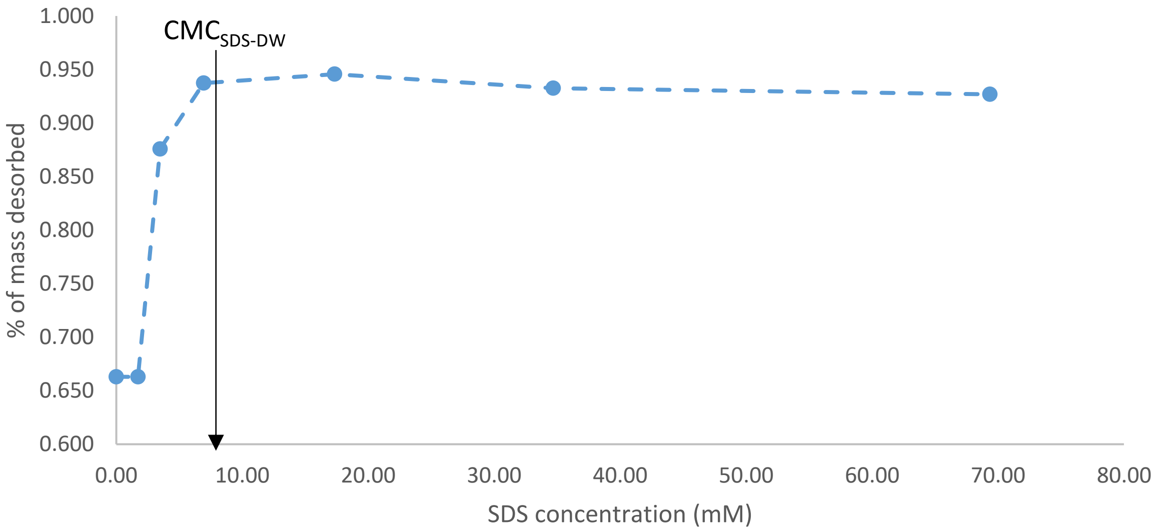


Figure 77: Graph showing the percentage of mass of crude oil B desorbed vs the SDS concentrations in distilled water

SDS - LS

Table 154: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by the low salinity solution and different SDS concentrations along with the appropriate percentages

SDS concentration (%)	average amount of crude oil B adsorbed (mg/cm^2)	Average amount of crude oil B desorbed (mg/cm^2)	average amount of crude oil B desorbed by LS (mg/cm^2)	average amount of crude oil B desorbed by SDS (mg/cm^2)	% of crude oil B removed by SDS	Total % of mass desorbed
0	0.0690	0.0485	0.0485	0	0.000	0.672
0.005	0.0969	0.0705	0.0651	0.00187	0.0193	0.691
0.010	0.118	0.101	0.0793	0.0216	0.183	0.855
0.020	0.143	0.131	0.0962	0.0349	0.244	0.916
0.050	0.133	0.124	0.0894	0.0348	0.262	0.934

0.100	0.107	0.100	0.0720	0.0282	0.263	0.935
0.200	0.138	0.130	0.0930	0.0368	0.266	0.938

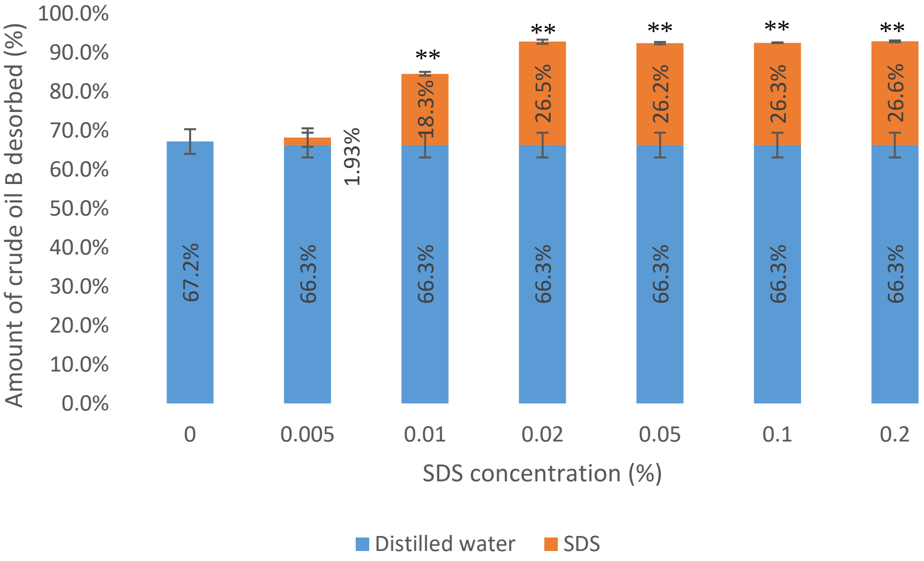


Figure 78: A histogram showing the percentage of crude oil B desorbed from the silica surface by low salinity and different SDS concentrations.

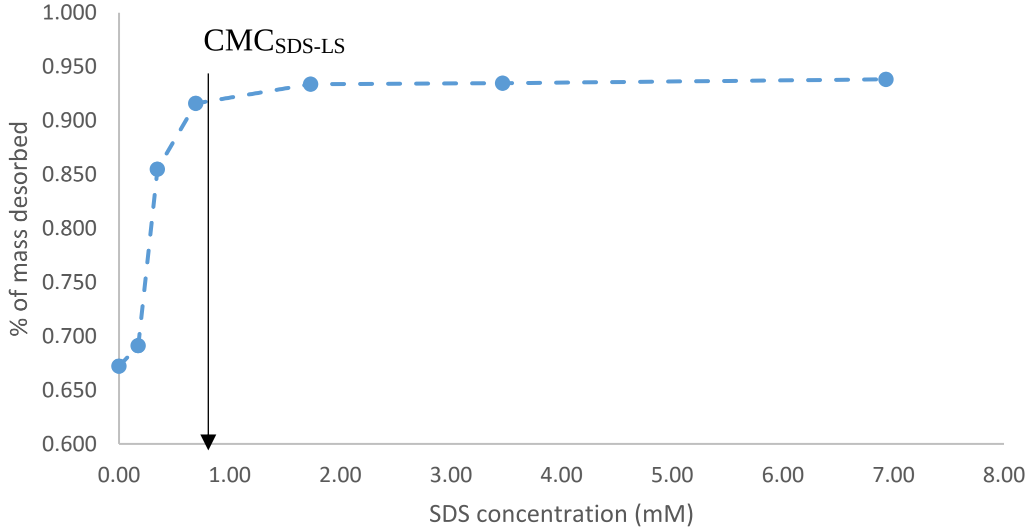


Figure 79: Graph showing the percentage of mass of crude oil B desorbed vs the SDS concentrations in low salinity

SDS – MS

Table 155: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by the moderate salinity solution and different SDS concentrations along with the appropriate percentages

SDS concentration (%)	average amount of crude oil B adsorbed (mg/cm^2)	Average amount of crude oil B desorbed (mg/cm^2)	average amount of crude oil B desorbed by MS (mg/cm^2)	average amount of crude oil B desorbed by SDS (mg/cm^2)	% of crude oil B removed by SDS	Total % of mass desorbed
0	0.1236	0.1034	0.1034	0	0.000	0.834
0.005	0.1087	0.0933	0.0907	0.00258	0.0238	0.858
0.010	0.116	0.104	0.0964	0.0079	0.069	0.903
0.020	0.111	0.101	0.0925	0.0085	0.076	0.911
0.050	0.131	0.122	0.1089	0.0129	0.099	0.933
0.100	0.125	0.117	0.1041	0.0124	0.099	0.934
0.200	0.112	0.105	0.0938	0.0118	0.105	0.939

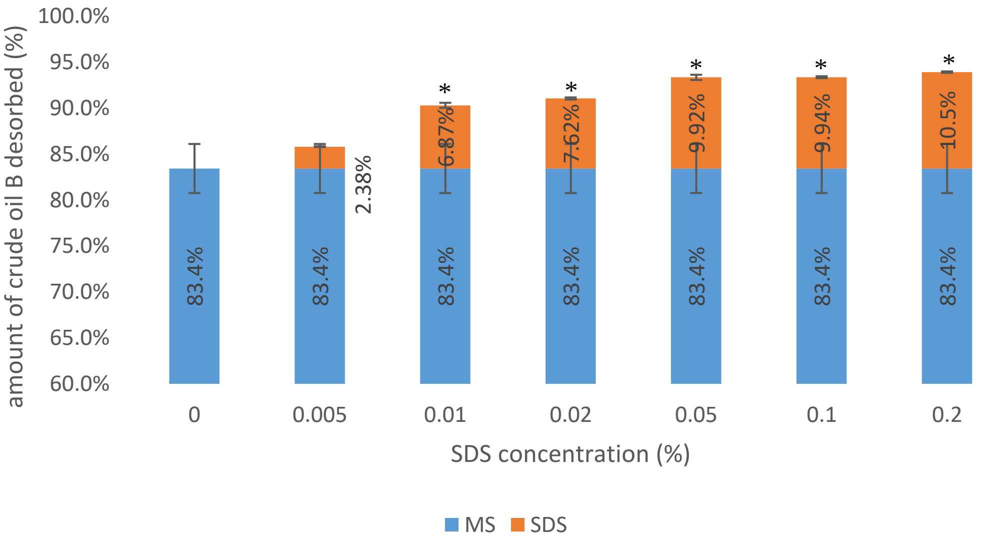


Figure 80: A histogram showing the percentage of crude oil B desorbed from the silica surface by moderate salinity solution and different SDS concentrations.

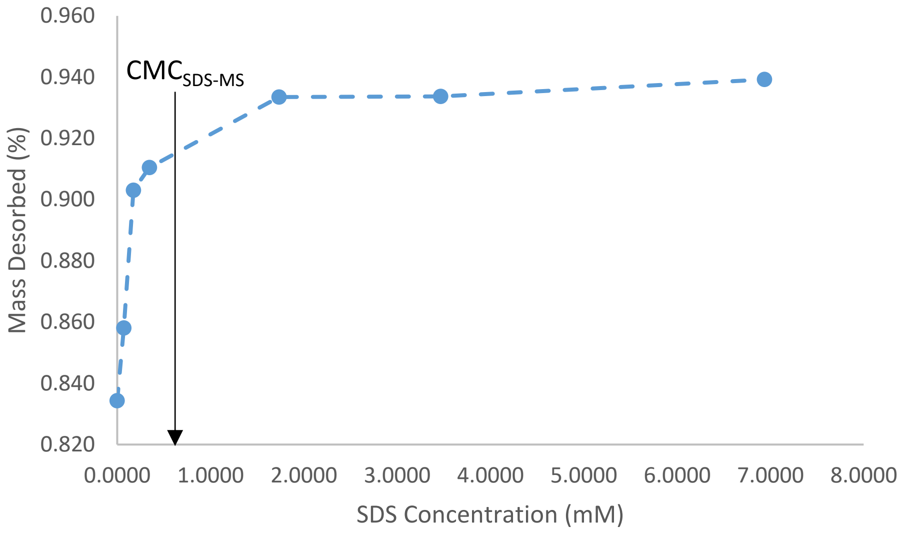


Figure 81: Graph showing the percentage of mass of crude oil B desorbed vs the SDS concentrations in moderate salinity

Triton X-100 - DW

Table 156: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by distilled water and different Triton X-100 concentrations along with the appropriate percentages

Triton X-100 concentration (%)	**average amount of crude oil B adsorbed (mg/cm^2)**	**Average amount of crude oil B desorbed (mg/cm^2)**	**average amount of crude oil B desorbed by DW (mg/cm^2)**	**average amount of crude oil B desorbed by Triton X-100 (mg/cm^2)**	**% of crude oil B removed by Triton X-100**	**Total % of mass desorbed**
0	0.0937	0.0619	0.0619	0	0.000	0.663
0.002	0.1262	0.106	0.0837	0.0224	0.178	0.841
0.005	0.0960	0.0838	0.0637	0.0195	0.203	0.866
0.010	0.114	0.102	0.0757	0.0259	0.226	0.889
0.015	0.0969	0.0847	0.0642	0.0202	0.208	0.871
0.020	0.119	0.104	0.0786	0.0249	0.210	0.873

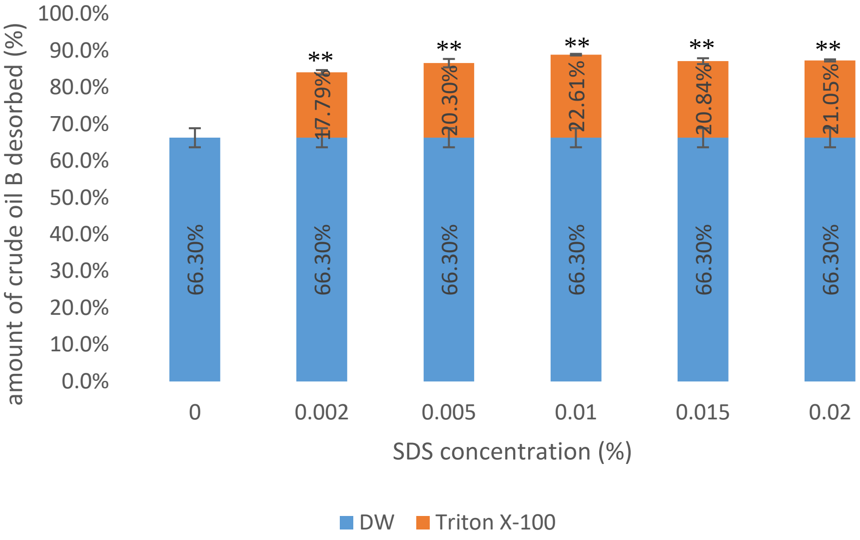


Figure 82: A histogram showing the percentage of crude oil B desorbed from the silica surface by distilled water and different Triton X-100 concentrations.

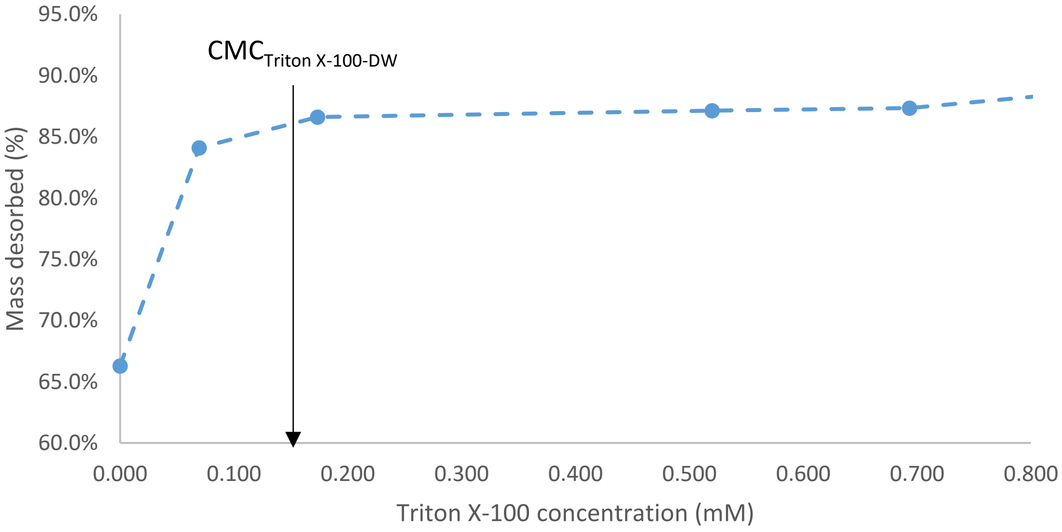


Figure 83: Graph showing the percentage of mass of crude oil B desorbed vs the Triton X-100 concentrations in distilled water

Triton X-100 - LS

Table 157: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by the low salinity solution and different Triton X-100 concentrations along with the appropriate percentages

Triton X-100 concentration (%)	**average amount of crude oil B adsorbed (mg/cm^2)**	**Average amount of crude oil B desorbed (mg/cm^2)**	**average amount of crude oil B desorbed by DW (mg/cm^2)**	**average amount of crude oil B desorbed by Triton X-100 (mg/cm^2)**	**% of crude oil B removed by Triton X-100**	**Total % of mass desorbed**
0	0.0690	0.0485	0.0485	0	0.000	0.672
0.001	0.0900	0.0798	0.0605	0.0188	0.209	0.881
0.002	0.117	0.106	0.0788	0.0276	0.236	0.908
0.005	0.0976	0.090	0.0656	0.0239	0.245	0.917
0.01	0.110	0.102	0.0741	0.0278	0.252	0.924
0.015	0.127	0.118	0.0853	0.0323	0.255	0.927
0.02	0.123	0.114	0.0824	0.0315	0.257	0.929

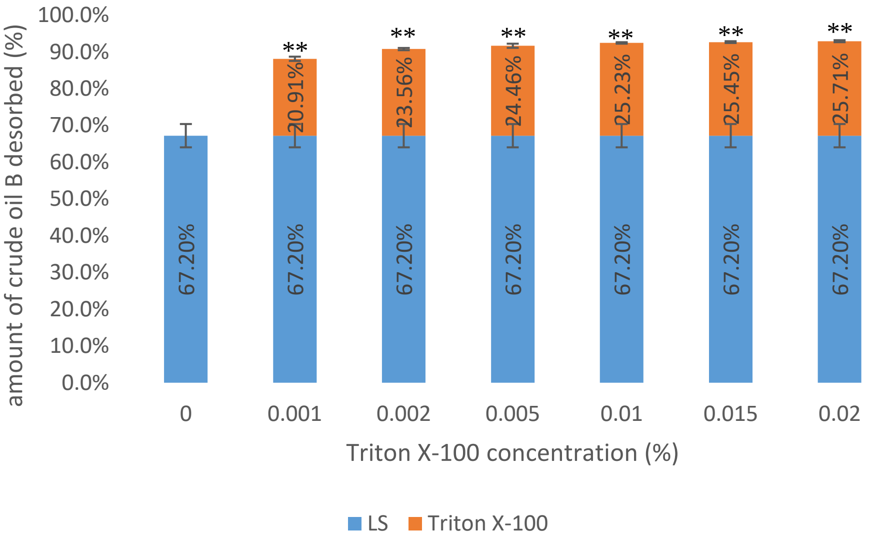


Figure 84: A histogram showing the percentage of crude oil B desorbed from the silica surface by low salinity solution and different Triton X-100 concentrations.

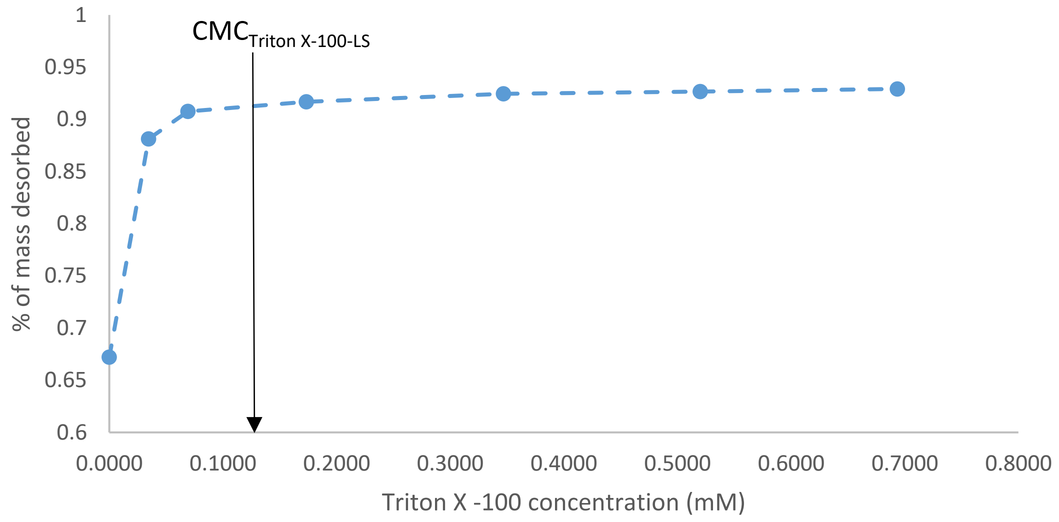


Figure 85: Graph showing the percentage of mass of crude oil B desorbed vs the Triton X-100 concentrations in low salinity.

Triton X-100 - MS

Table 158: The total average amount of crude oil B adsorbed and desorbed from the silica surface, the amount removed by moderate salinity and different Triton X-100 concentrations along with the appropriate percentages

Triton X-100 concentration (%)	**average amount of crude oil B adsorbed (mg/cm^2)**	**Average amount of crude oil B desorbed (mg/cm^2)**	**average amount of crude oil B desorbed by DW (mg/cm^2)**	**average amount of crude oil B desorbed by Triton X-10 (mg/cm^2)**	**% of crude oil B removed by Triton X-100**	**Total % of mass desorbed**
0	0.1236	0.1034	0.1034	0	0.000	0.834
0.001	0.0955	0.0852	0.0797	0.00547	0.0573	0.892
0.0015	0.113	0.102	0.0943	0.0078	0.069	0.904
0.002	0.115	0.104	0.0959	0.0085	0.074	0.908
0.005	0.124	0.113	0.1033	0.0093	0.075	0.910
0.01	0.121	0.110	0.1010	0.0092	0.076	0.911
0.015	0.098	0.090	0.0814	0.0083	0.085	0.920

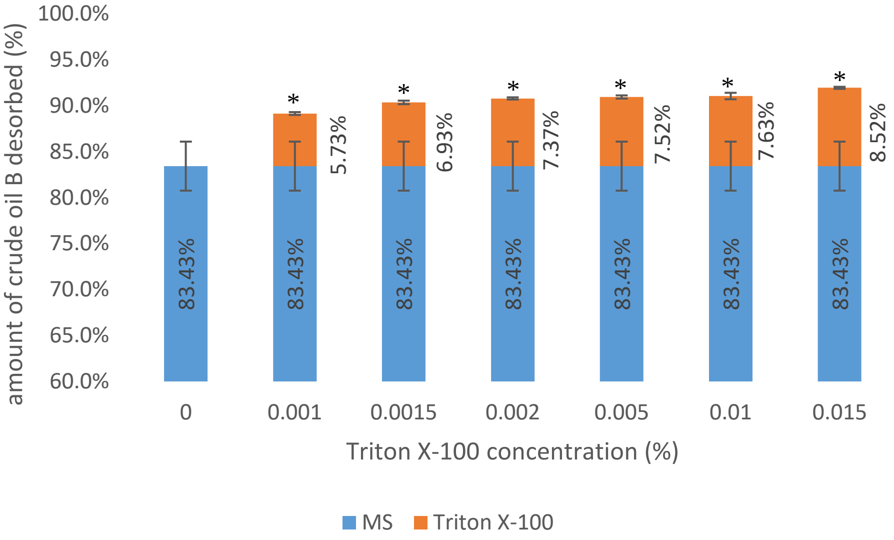


Figure 86: A histogram showing the percentage of crude oil B desorbed from the silica surface by moderate salinity solution and different Triton X-100 concentrations.

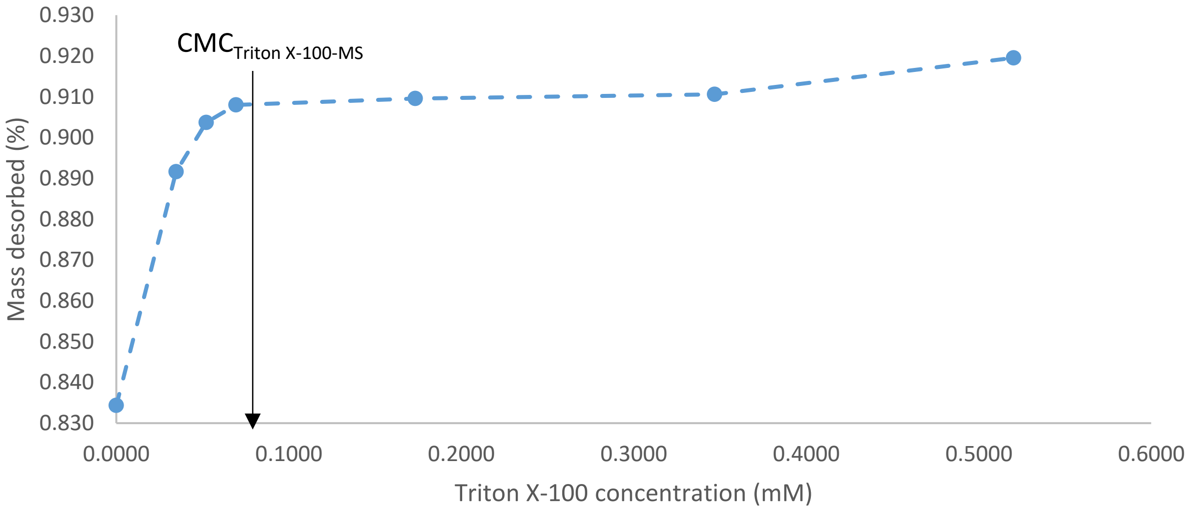


Figure 87: Graph showing the percentage of mass of crude oil B desorbed vs the Triton X-100 concentrations in moderate salinity

2. *Crude oil A*

For each run, different concentrations of surfactant were injected simulteaneously where the control solution is being introduced before each injection. The amount of crude oil A removed is is obtained after each injection of a surfactant solution. Therefore, the cumulative mass desorbed for each run was normalized to the total amount of oil adsorbed to the surface. The average of the mass desorbed by each SDS concentration of the three runs is plotted vs the SDS concentrations to determine how the addition of surfactant can change the amount of oil desorbed.

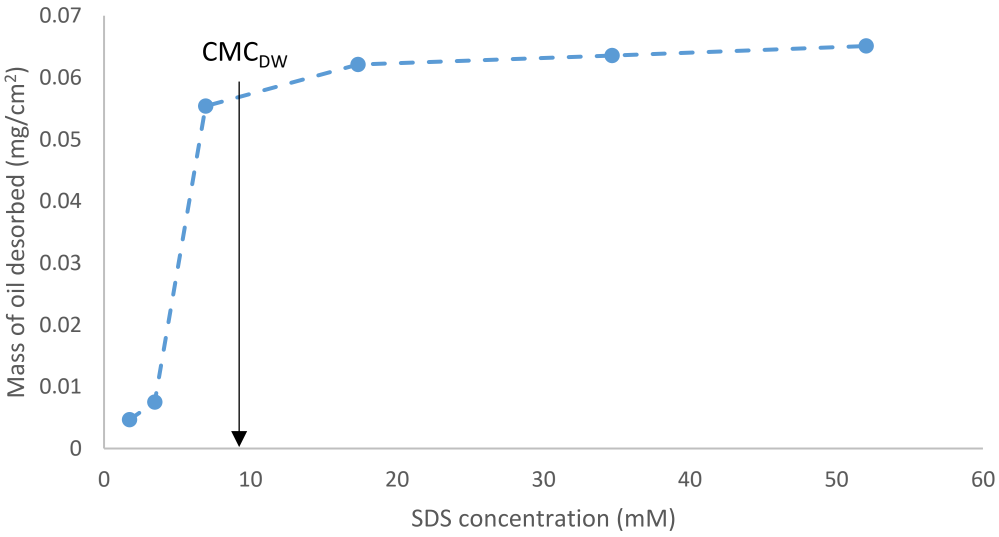


Figure 88: The amount of crude oil A desorbed from the silica sensor vs the change in SDS concentration in distilled water.

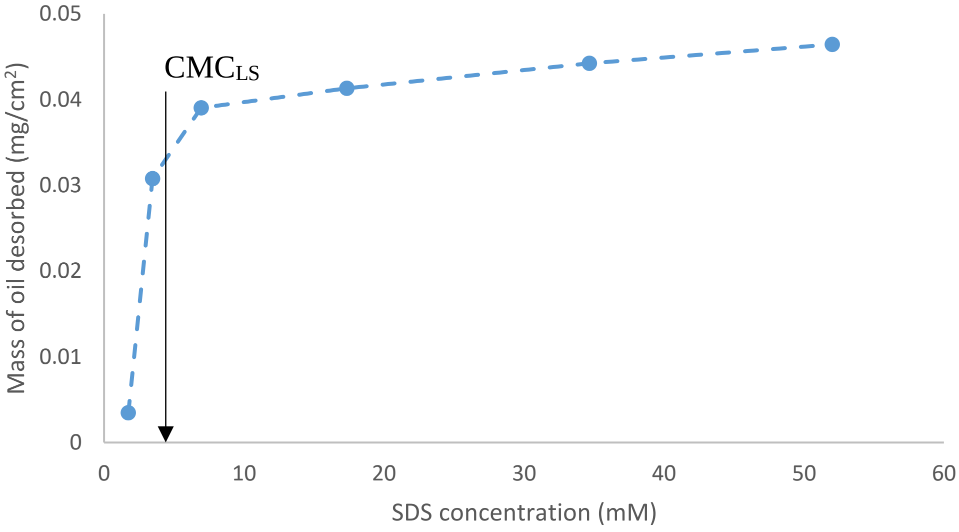


Figure 89: The amount of crude oil A desorbed from the silica sensor vs the change in SDS concentration in low salinity.

The amount of crude oil B adsorbed on the silica surface is observed to be in the range of 0.0690-0.143 mg/cm^2 while the amount of crude oil A adsorbed on the silica surface was determined to be around 0.0276 mg/cm^2.

As we look at each graph for the two surfactants, we realize that as we increase the concentration of surfactant the amount of oil desorbed from the surface increases until reaching a certain value that is the CMC. After this point, the amount of oil stabilizes or converges to a certain value. When the concentration of surfactant is increased, the oil-water interfacial tension will be reduced, as well as the oil-mineral interface. In this way, just below the CMC, the particles of oil that are desorbed from the surface reach a certain maximum where no more desorption occurs upon adding more surfactants. This stabilization is due to the formation of micelles which limits the desorption process from the surface. If we take SDS in distilled water as an example, we realize that the injection of

0.2% SDS in distilled water has led to a maximum desorption keeping note that the CMC of SDS solution in distilled water is around 0.239% (8.29mM). At this point, the average amount of crude oil B desorbed is around 0.0360 mg/cm^2, which is approximately 27.4% of crude oil B removal by this SDS concentration. These results are confirmed with the crude oil A where the maximum amount of oil is desorbed after injecting 0.2% SDS in distilled water.

For the Triton X-100 surfactant in distilled water, the CMC is at much lower concentrations and about 0.0136% (0.211mM). The maximum amount of crude oil B removal occurred at 0.01% Triton X-100. At this concentration, 0.0258 mg/cm^2 of crude oil B was desorbed on average leading to around 22.6% of removal by the surfactant.
Upon adding more surfactant, the interfacial tension between the oil and water is minimized to a certain extent causing the force of adhesion between the mineral and the crude oil to be reduced.

In addition, the change in dissipation can also determine the stiffness of the surface. If there is an increase in dissipation, it means that a thin film has been formed on the surface while a decrease in the dissipation means a deformation of this layer. For the different concentrations of surfactants mentioned earlier, the percentage change in dissipation was shown to be the highest. This confirm that at these concentrations, the biggest portion of the crude oil layer is being removed.

B. Effect of salinity.

Salinity has a major influence on the formation of microemulsions and this is shown from the change of the CMC value for the two surfactants after addition of sodium chloride. As shown in figure 90, the CMC of SDS has decreased from 8.29 mM to 2.99 mM and 1.40 mM with low and moderate salinity solutions respectively. This trend was also seen with Triton X-100 surfactant where the CMC has decreased from 0.211 mM to 0.135 mM with low salinity and 0.0727 mM with moderate salinity.

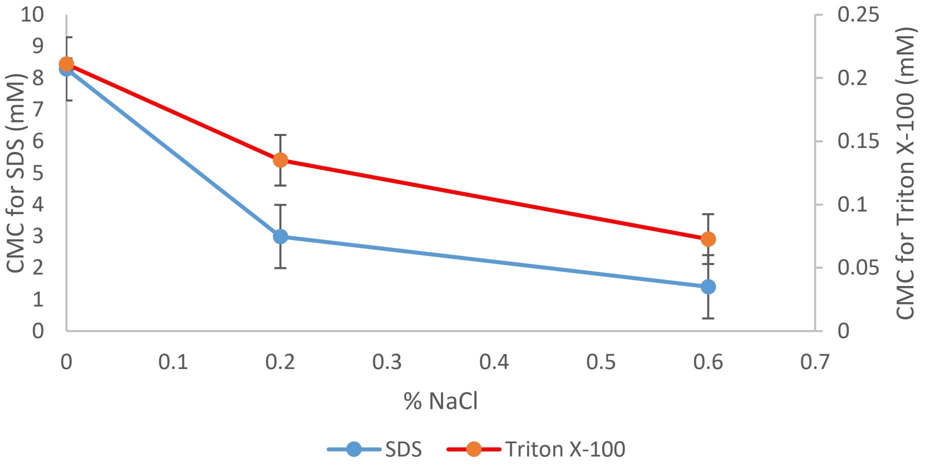


Figure 90: Change of CMC values for SDS and Triton X-100 as the amount of electrolyte increase in the solution.

Once the CMC has decreased upon addition of salty solutions, the formation of microemulsion appears at lower concentration of surfactant. Hence this will lead to the maximum desorption of crude oil at lower values. For the SDS surfactant, the CMC values are marked in the graphs below with crosses: blue for DW, red for LS and purple for MS.

For both crude oil A and crude oil B, the amount of oil desorbed from the surface has reached a maximum at lower values as we increase the concentration of sodium chloride.

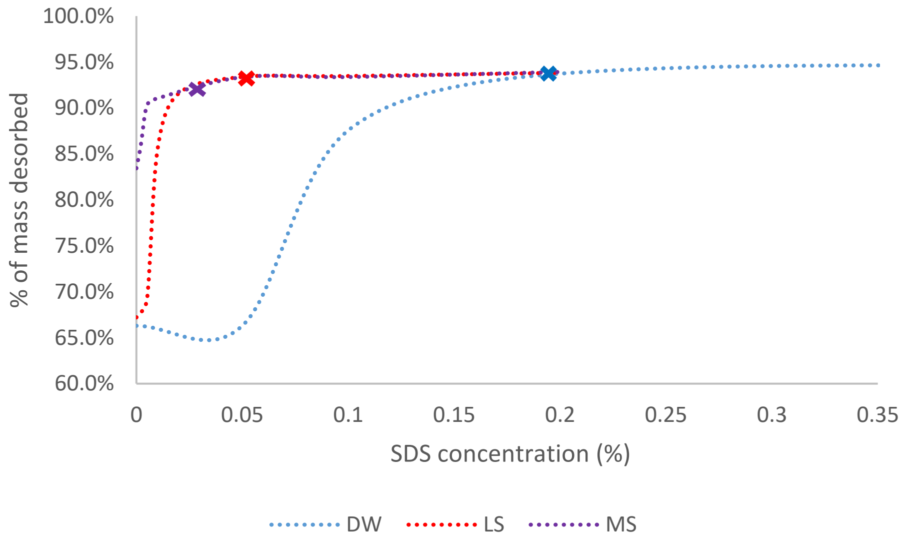


Figure 91: The percentage of crude oil B mass desorbed vs the SDS concentrations for distilled water, low salinity and moderate salinity

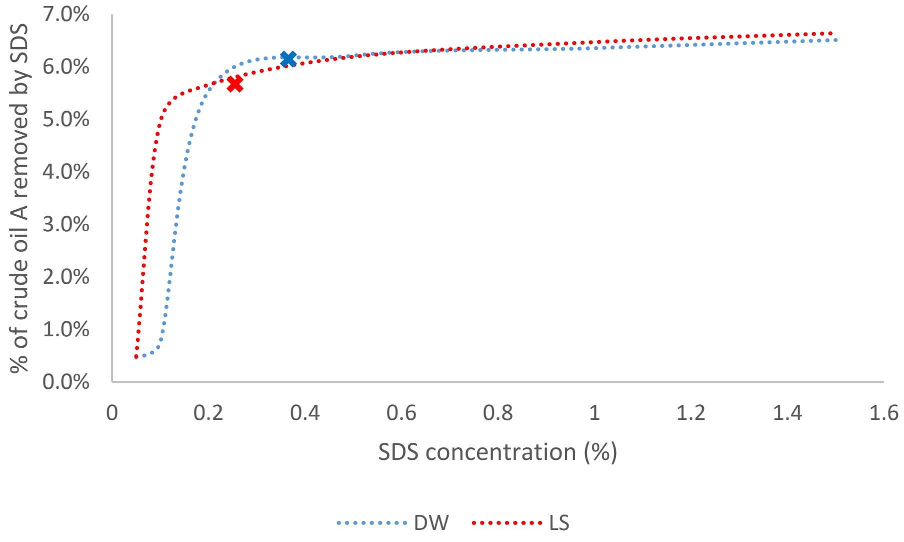


Figure 92: The percentage of crude oil A mass desorbed vs the SDS concentrations for distilled water and low salinity

However, the performance of SDS on the two types of crude oil did not improve by much after the addition of saline solution. For the crude oil B shown in figure 91, the total percentage of crude oil B desorbed from the silica surface has increase from 92.7% with distilled water to 93.8% and 93.9% in the presence of low and moderate salinity respectively. For crude oil A shown in figure 92**,** the percentage of oil removed by SDS from the silica sensor has barely increased from a value of 6.51% with distilled water until 6.64% with low salinity solution.

Comparing the three control solutions where Triton X-100 is the surfactant involved has led to different results. The stars placed on the graph represent the different CMC values where the blue is for distilled water, the red is for low salinity and the purple is for moderate salinity. As the salinity increases, the CMC values decrease correspondingly and the percentage of crude oil B desorbed has reached a maximum at lower Triton X-100 concentrations. Upon the addition of sodium chloride, more of the crude oil B has been desorbed from the surface. The percentage of crude oil B desorbed has reached a value of 92.9% instead of 87.3% for distilled water. The presence of electrolyte with the nonionic surfactant has enhanced the desorption of the oil from the silica surface.

Higher concentration of NaCl couldn't be considered in order to mimic brine solutions. Using high salinity solutions has lead to formation of precipitation due to the Kraft temperature of the surfactants studied.

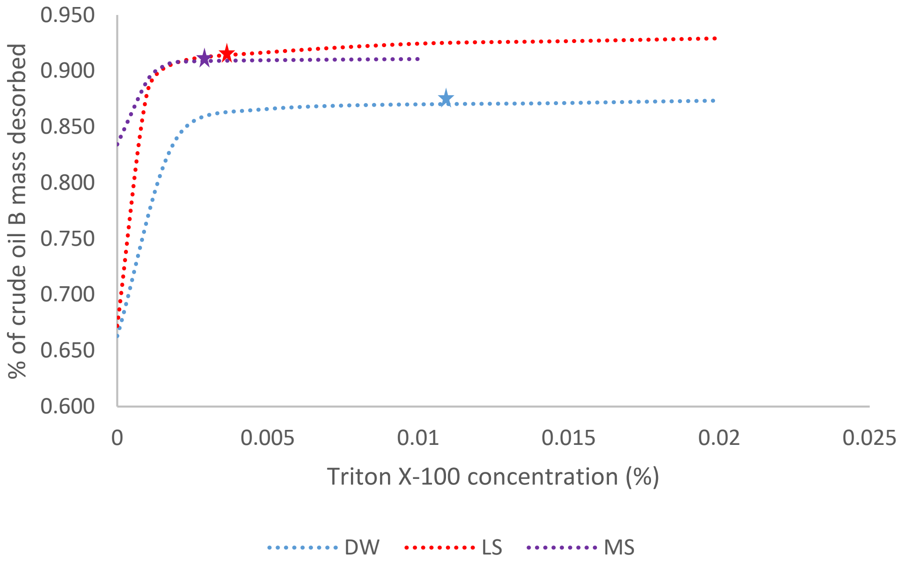


Figure 93: The percentage of crude oil B mass desorbed vs Triton X-100 concentrations for distilled water, low salinity and moderate salinity

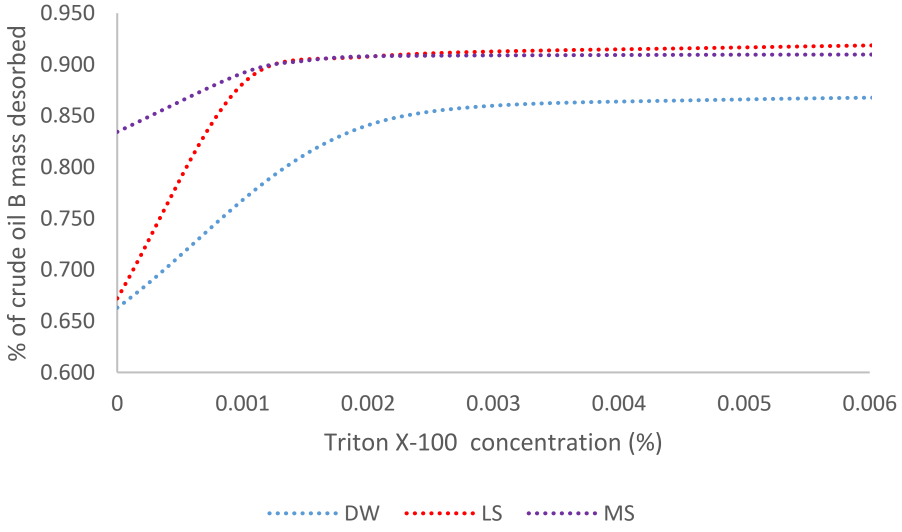


Figure 94: Zoomed section of the graph representing the percentage of crude oil B mass desorbed vs Triton X-100 concentrations for distilled water, low salinity and moderate salinity

C. Effect of surfactant type

Two different surfactant types were studied: an anionic and nonionic surfactant. This change of charges may affects the desorption process of oil on the silica solution which is an ionic surface.

The results mentioned are obtained with the washing effect using a control solution. As presented in figure 95, the amount of oil desorbed by Triton X-100 is lower than the amount desorbed by SDS. The value has increased from 87.3% to 92.7% using Triton X-100 and SDS respectively.

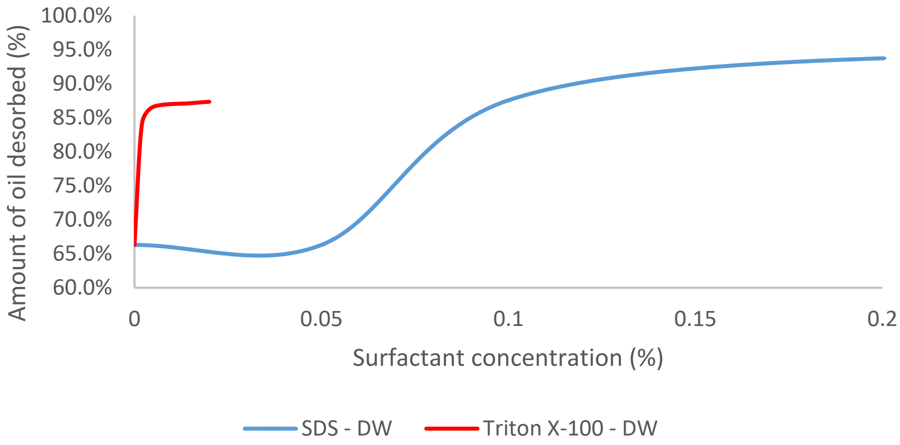


Figure 95: The amount of oil desorbed from the silica surface by SDS and Triton X-100 in distilled water.

These results were also obtained for SDS and Triton X-100 in moderate salinity, but with less effect than in the distilled water. The percentage has increased from 91.1% to 93.4% using Triton X-100 and SDS respectively as shown in figure 96.

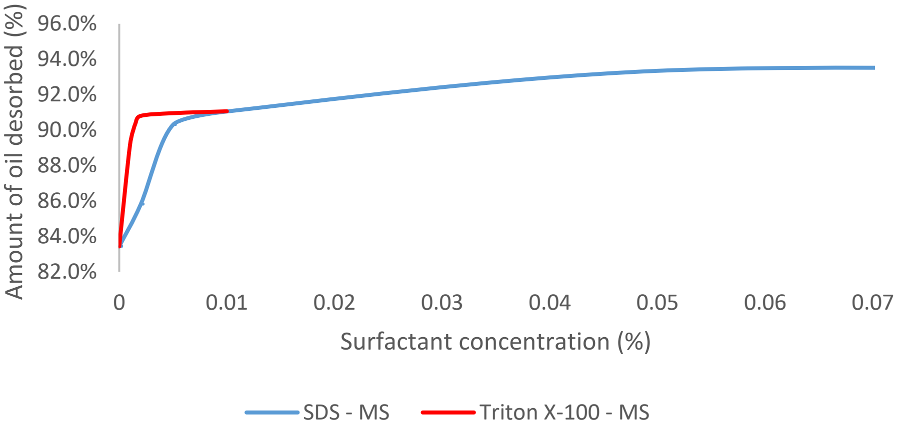


Figure 96: The amount of oil desorbed from the silica surface by SDS and Triton X-100 in moderate salinity.

However, with the low salinity solutions, there is no difference between the two types of surfactants: almost the same results were obtained with a value of 92.9% using SDS and 93.8% using Triton X-100.

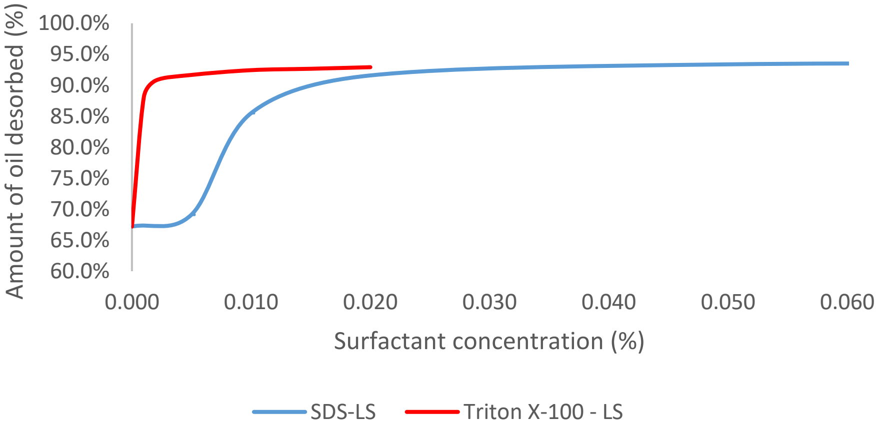


Figure 97: The amount of oil desorbed from the silica surface by SDS and Triton X-100 in low salinity.

There is no apparent trend shown when increasing the salinity of the solutions. This may be due to experimental error.

D. Effect of temperature

One of the objectives in this research was to study the effect of temperature on the desorption of crude oil from the surface of silica sensor. However, using the QCM-D, we failed to meet this objective as fluctuations were observed for the frequency change. The graph below shows how the frequency is being fluctuated at a temperature equal to 35ºC.

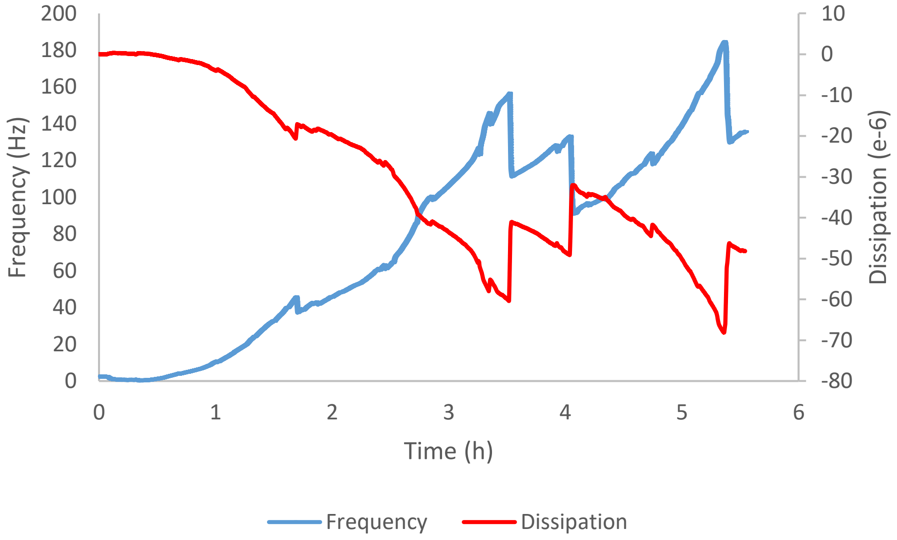


Figure 98: A representative graph showing the change in frequency and dissipation at T=35^{O}C

CHAPTER V

FUTURE WORK

Without any doubt, the experimental work should proceed for the fundamental future development in this field.

The QCM-D is an important tool in determining the adsorption and desorption of material on and off the surface. Studying the chemical enhanced oil recovery process in miniature scale by mimicking the reservoir should be developed further by examining the effect of temperature on the behavior of different surfactants. In addition, different mineral surfaces other than the silica surface should be considered in order to mimic other types of reservoirs such as carbonate reservoirs.

More experiments should be performed with much lower concentrations of Triton X-100 to be able to show a full picture of the effect of this surfactant's concentration on the desorption process.

In addition, the same experiments done with crude oil B can be done with crude oil A and vice versa. In this way, we can study how different types of oil can change the desorption rate on the mineral surface using the same surfactant.

Moreover, a cationic surfactant can also be examined to compare the effect of the surfactant's charge or studying surfactants with the same charge but changing their molecular structure i.e. their tail in order to study the effect of the hydrophobic part.

In this manner, there is always ongoing research about his topic using the QCM-D technique where surfactants, salinities, temperature, minerals, etc. can be altered in order to determine the optimal combination for Enhanced Oil Recovery.

Another technique may be considered where adsorption columns filled with the appropriate mineral can be used in order to study the adsorption of surfactants on the mineral surface.

CHAPTER VI

CONCLUSION

During the last decades, the discovery of oil was declining making place for chemical EOR, which holds a promise for future enhancement in oil production. The use of surfactants as a chemical EOR showed encouraging results, but the need for high concentration limited its use in the mid-1980s. The change of wettability due to the induction of surfactant has been intensely considered in the past 50 years as a way to reduce the oil saturation present in the rock of reservoir. This method that help in increasing the oil recovery continues to undertake laboratory analysis.

The main purpose of this project was to investigate the effect of two surfactants: SDS and Triton X-100 as agents for enhanced oil recovery using the QCM-D technique. Different parameters such as concentrations, salinity and temperatures where altered.
One light crude oil (crude oil A) and one moderately heavy crude oil (crude oil B) were used for the desorption process. As the concentration of surfactant increases, more crude oil B has been desorbed on the silica surface with a maximum desorption around the CMC of 27.4% and 22.6% using SDS and TritonX-100 respectively, in distilled water.

Increasing the salinity of the control solution has decreased the CMC of two surfactants leading to reaching the maximum desorption by a smaller amount of surfactant. SDS did not help in reaching higher amount of desorption however, with Triton X-100, the total percentage of oil desorption has increased from 87.3% to 92.9%.

A major difficulty faced was to increase the temperature using the QCM-D: fluctuations were observed at 35ºC and 50ºC. Therefore, the study of temperature was eliminated at this stage.

CHAPTER VII

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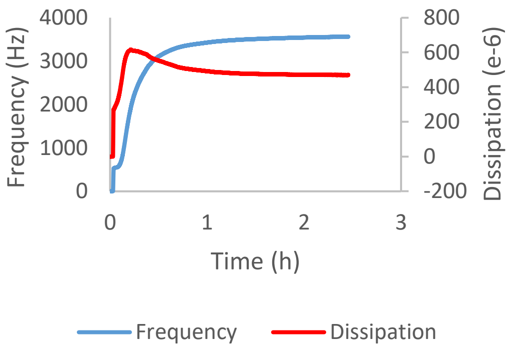
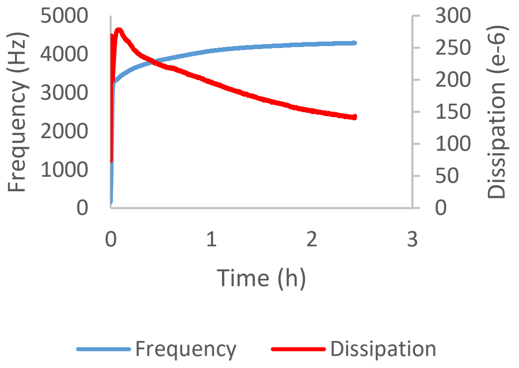
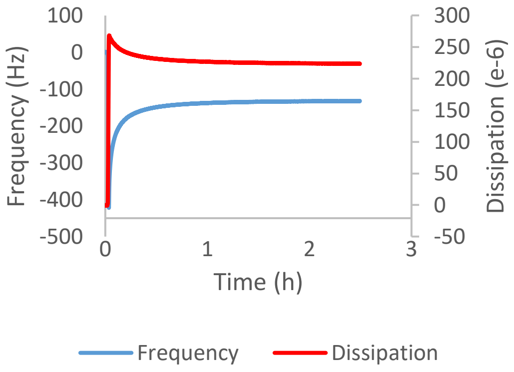
APPENDIX A

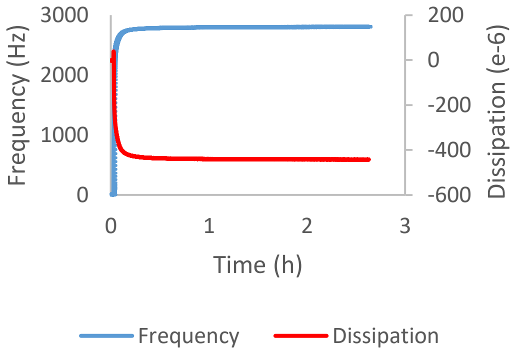
- DW



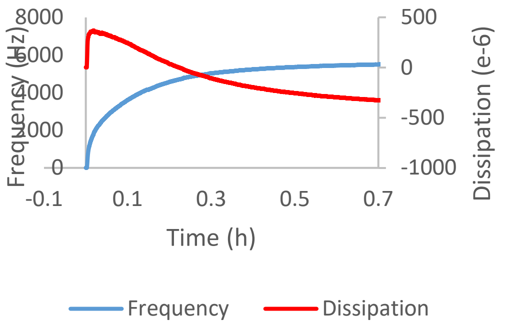


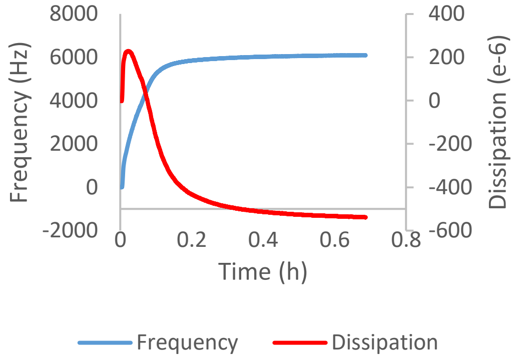
- LS



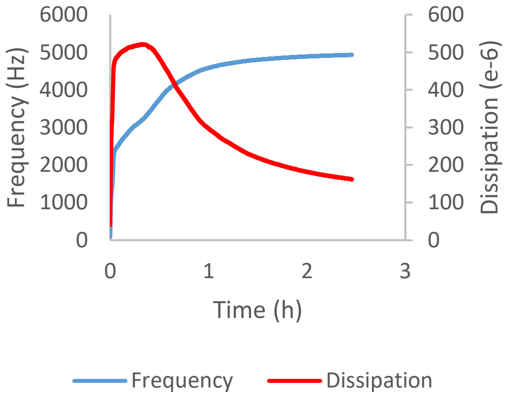


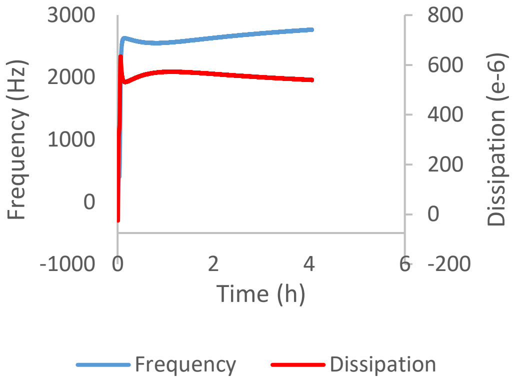
- MS



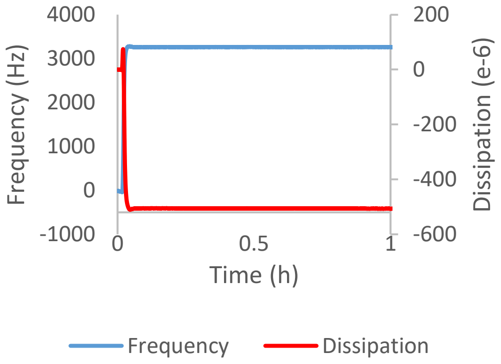


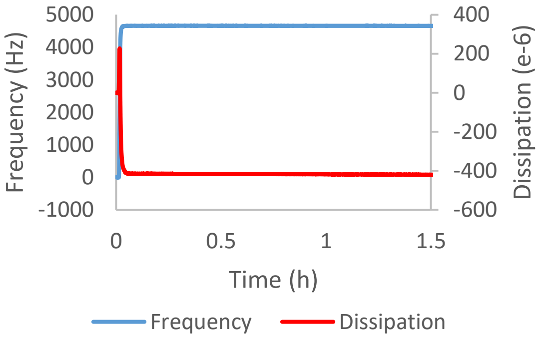
- 0.05% SDS – DW



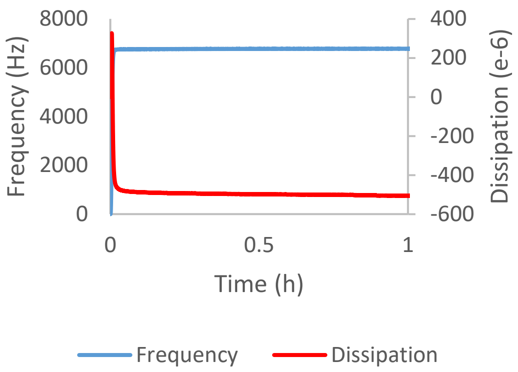


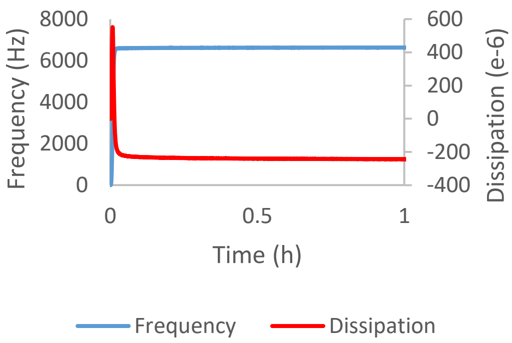
- 0.1% SDS – DW



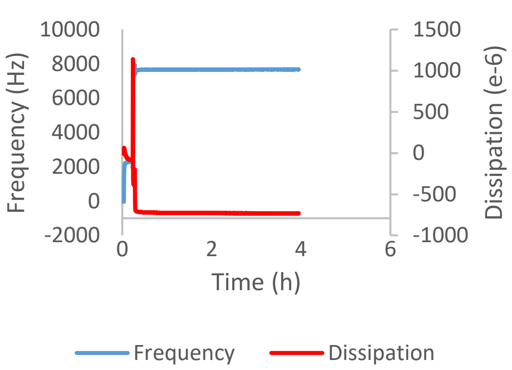


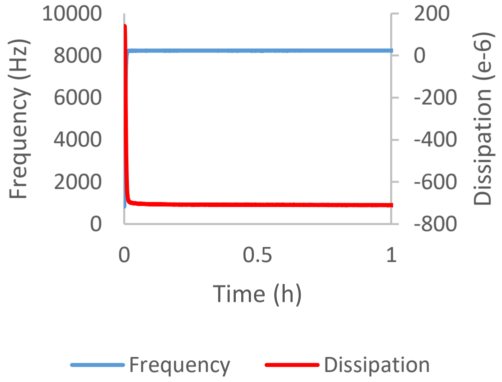
- 0.2% SDS – DW



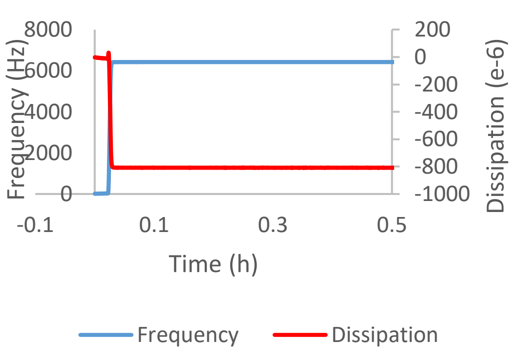


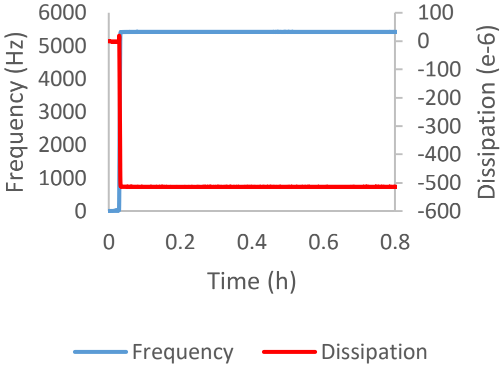
- 0.5% SDS – DW



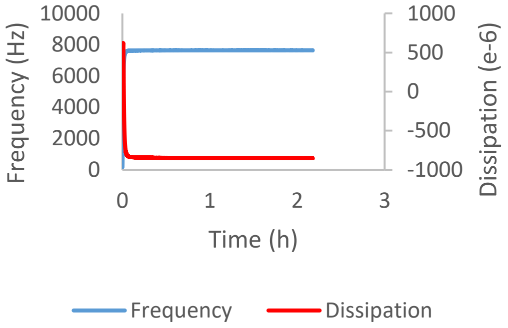


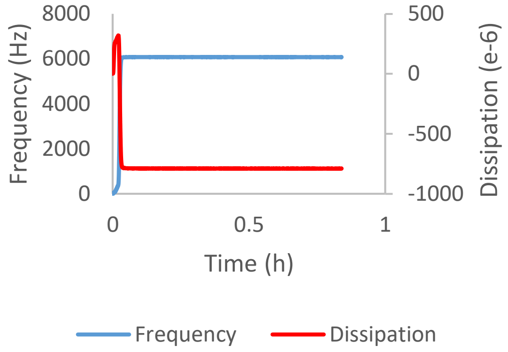
- 1% SDS – DW



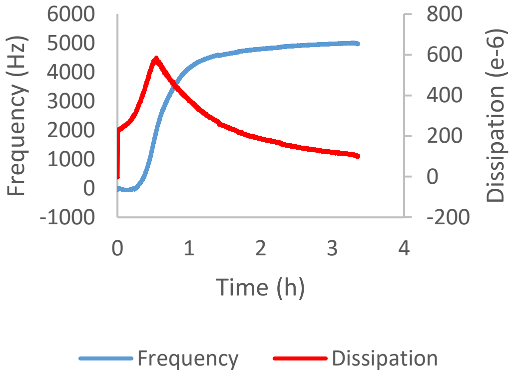


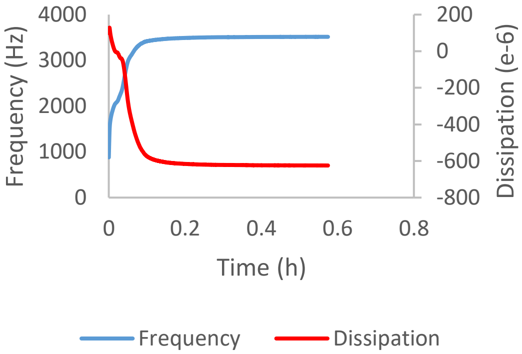
- 2% SDS – DW



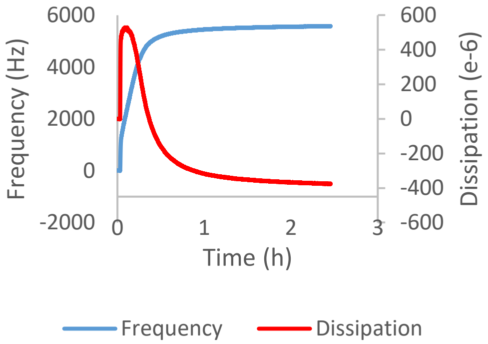


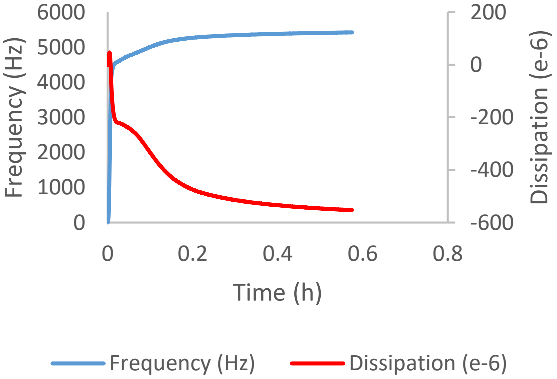
- 0.005% SDS – LS



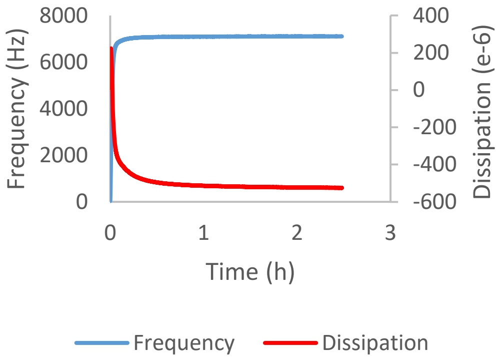


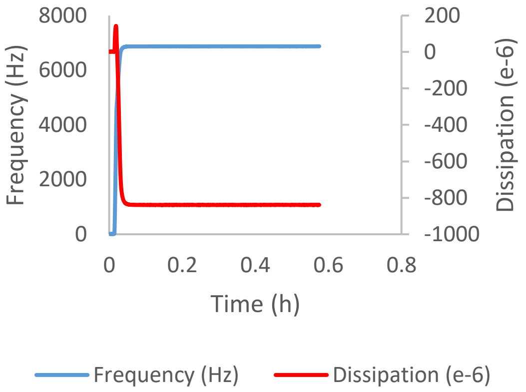
- 0.01% SDS – LS



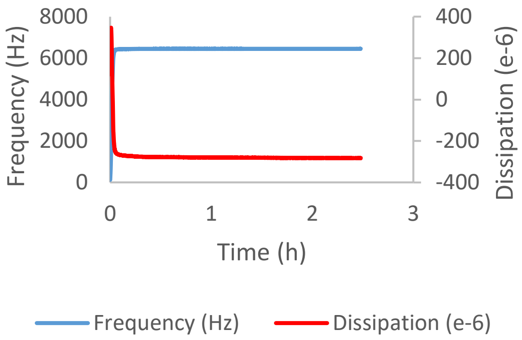


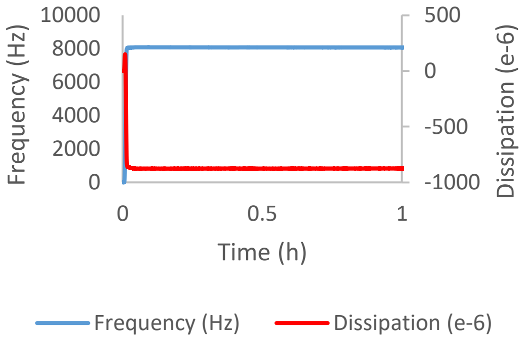
- 0.02% SDS – LS



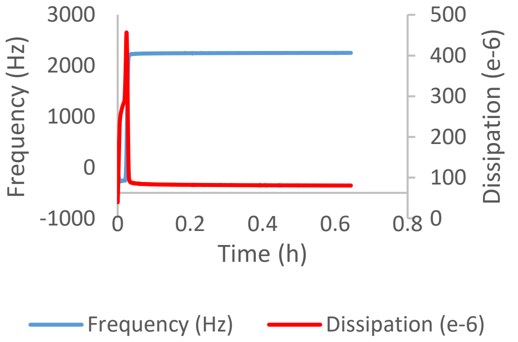


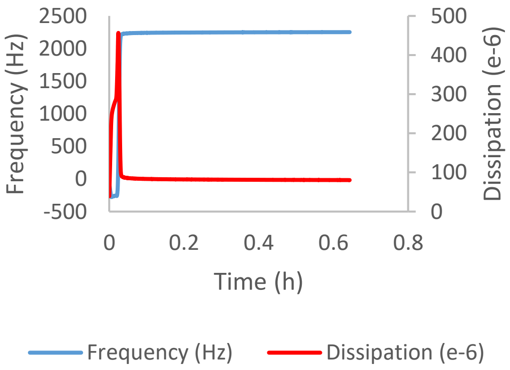
- 0.05% SDS – LS



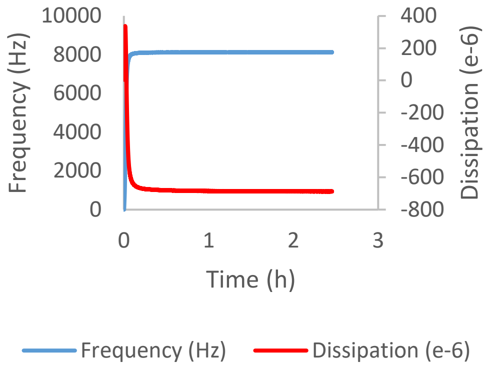


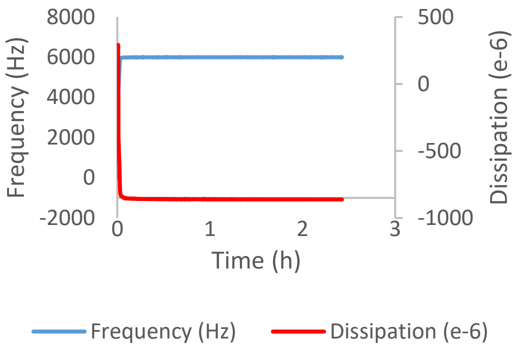
- 0.1% SDS – LS





- 0.2% SDS – LS





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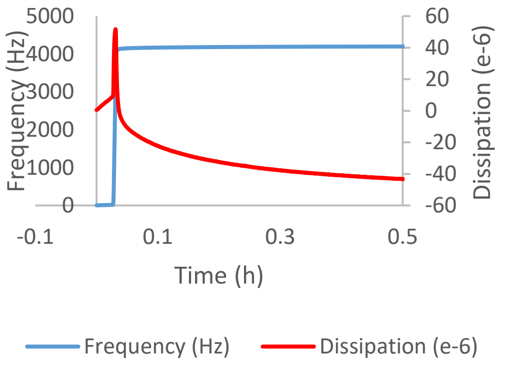

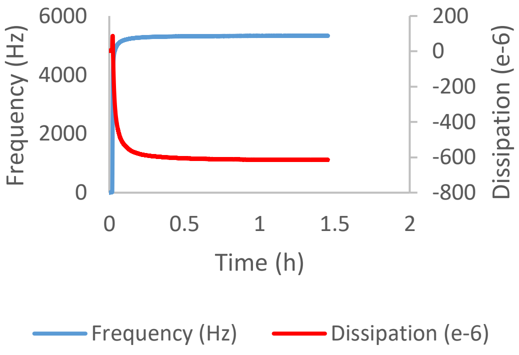

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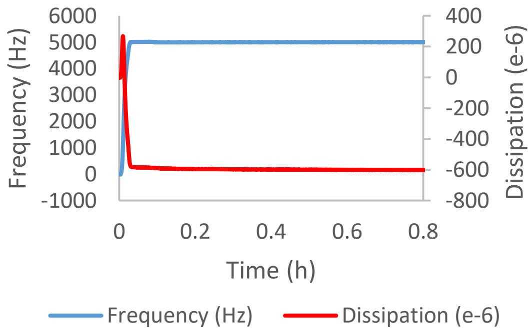
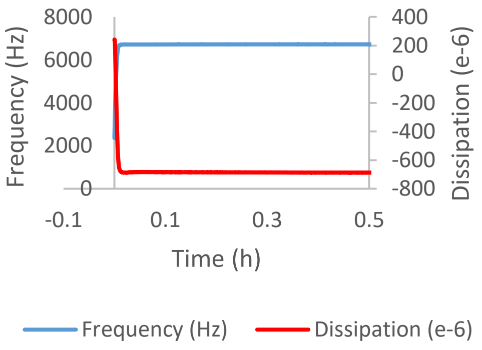
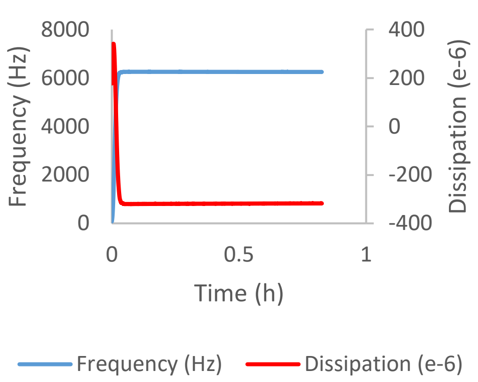
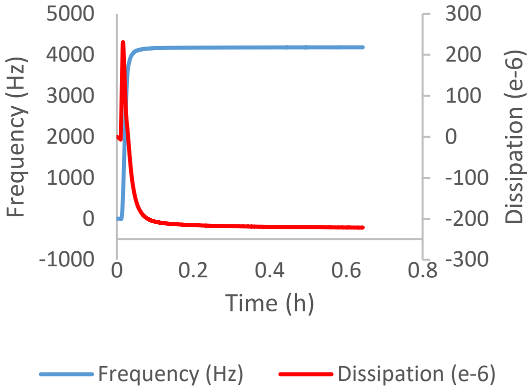




- 0.01% SDS – MS





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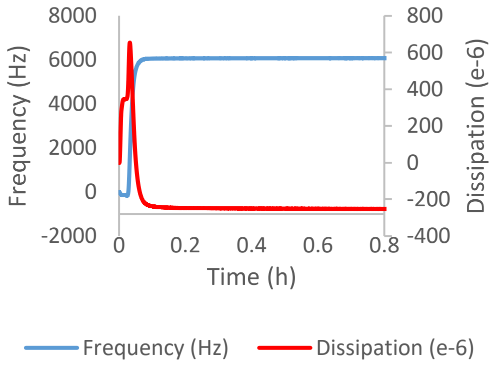
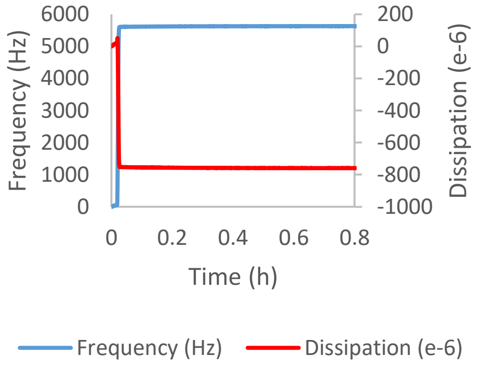
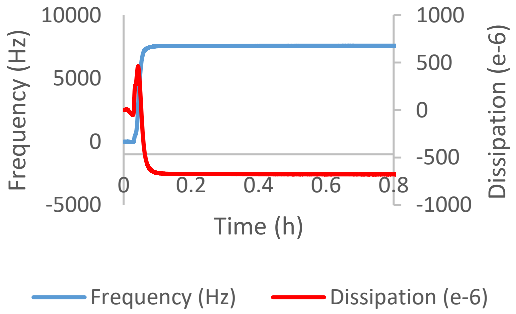
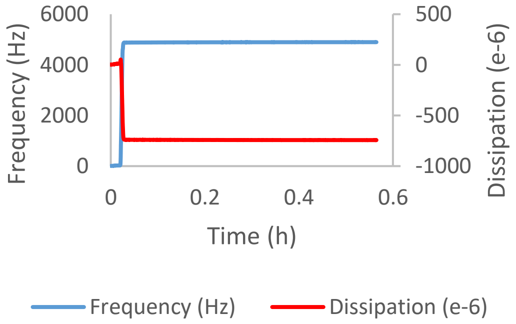


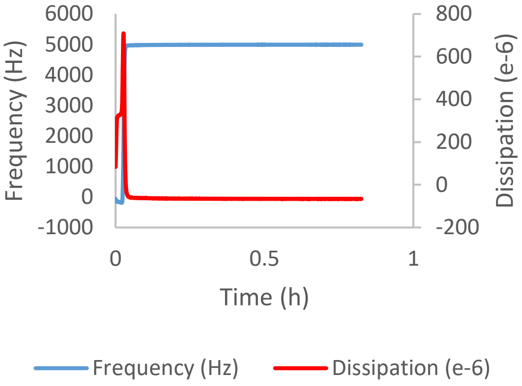


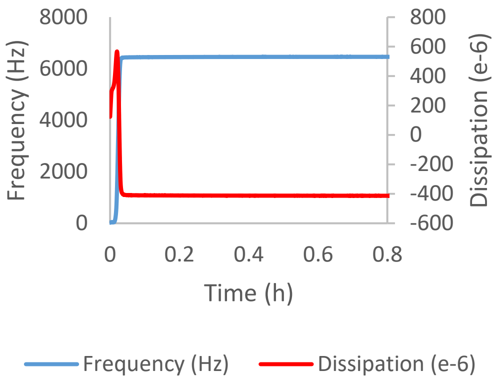
- 0.1% SDS – MS



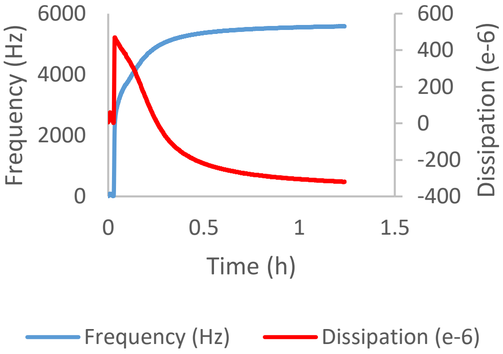


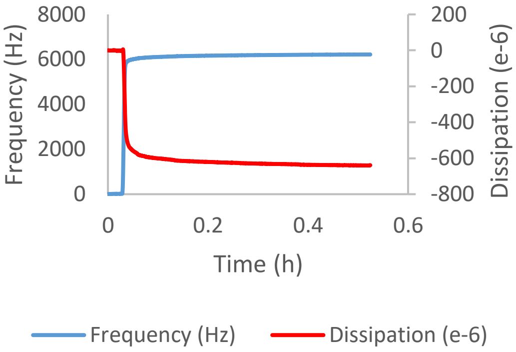
- 0.2% SDS – MS



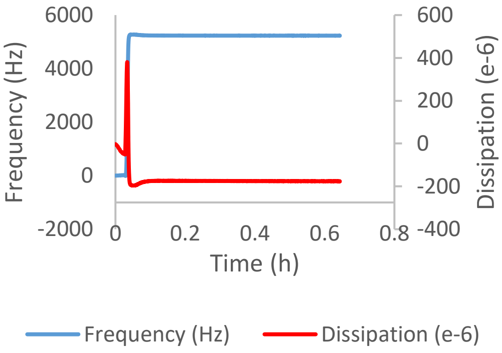


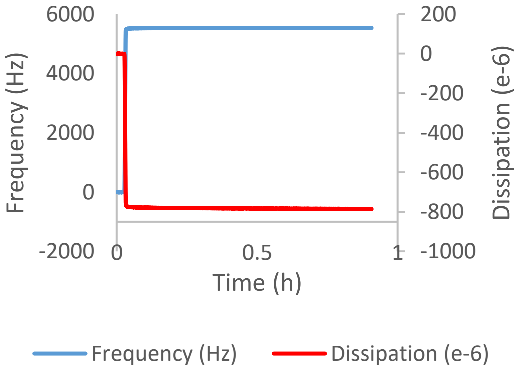
- 0.002% Triton X-100 – DW



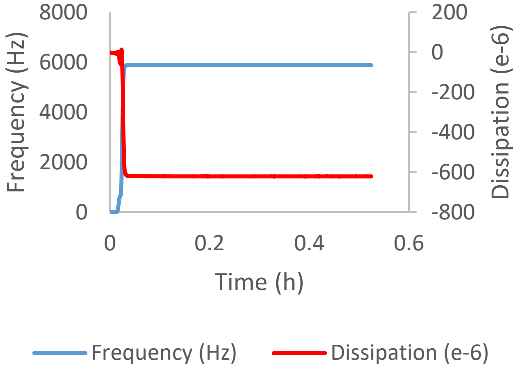


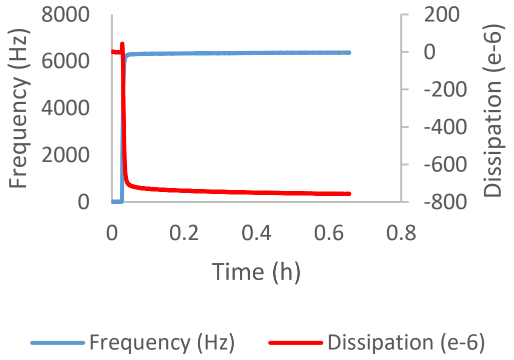
- 0.005% Triton X-100 – DW



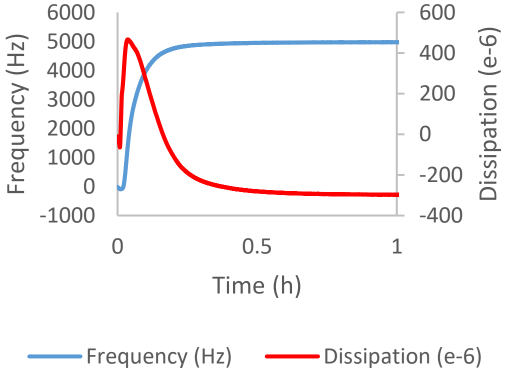


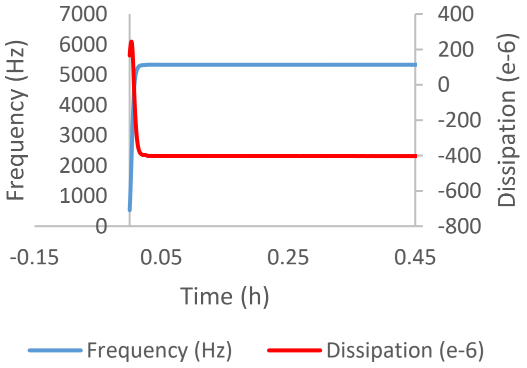
- 0.01% Triton X-100 – DW



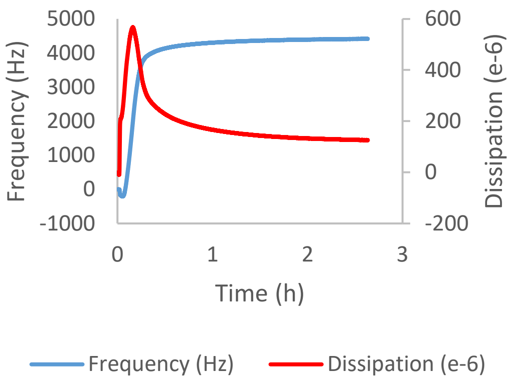


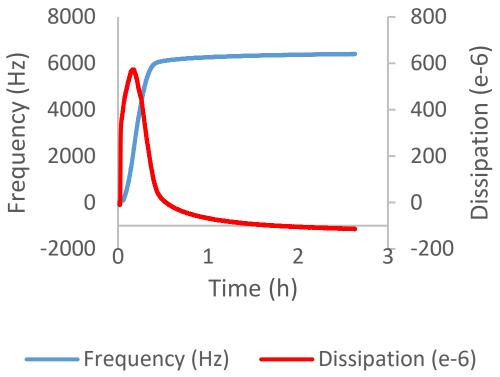
- 0.015% Triton X-100 – DW



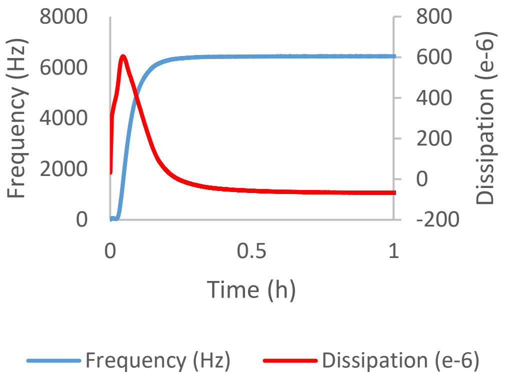


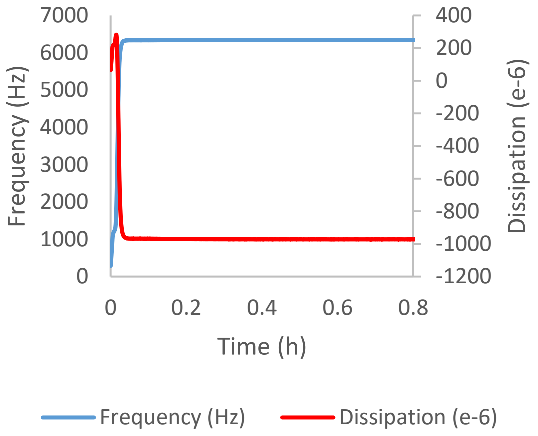
- 0.02% Triton X-100 – DW



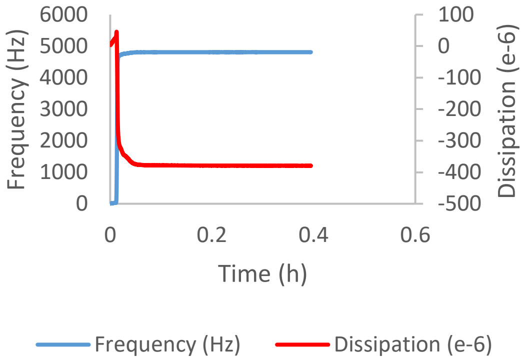


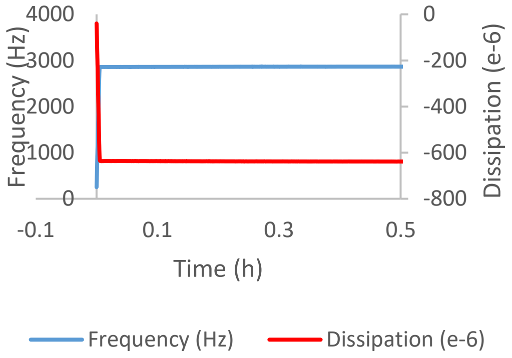
- 0.04% Triton X-100 – DW



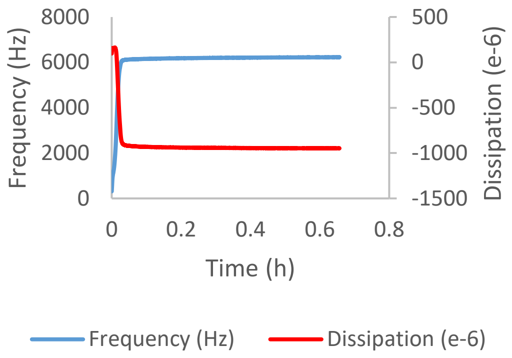


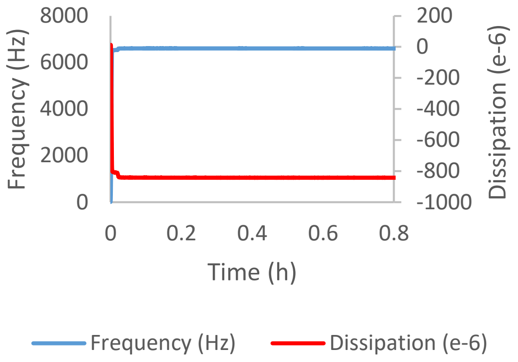
- 0.001% Triton X-100 – LS



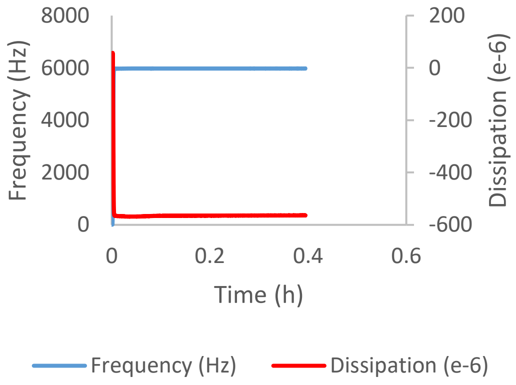


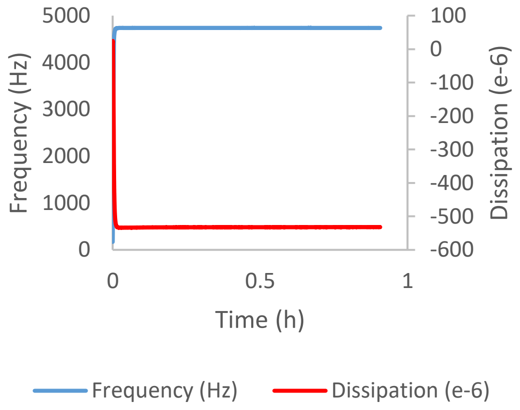
- 0.002% Triton X-100 – LS



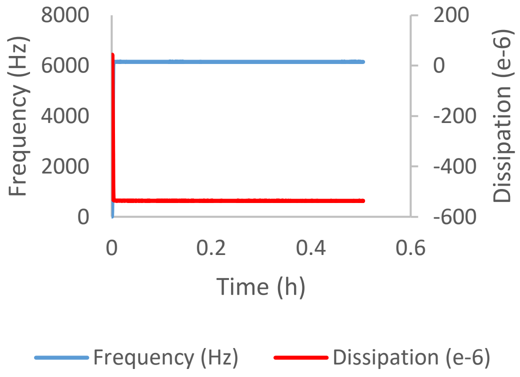


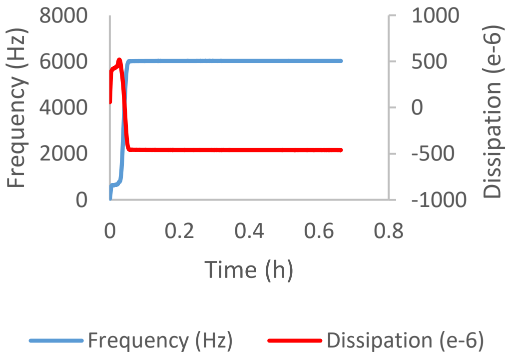
- 0.005% Triton X-100 – LS



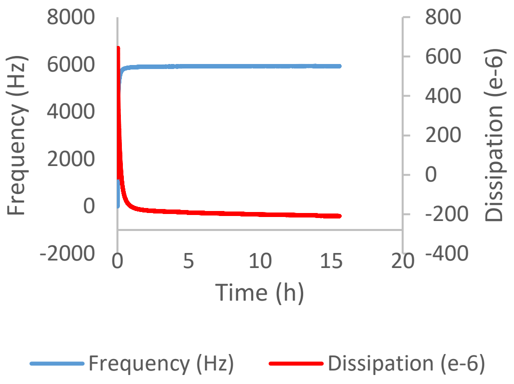


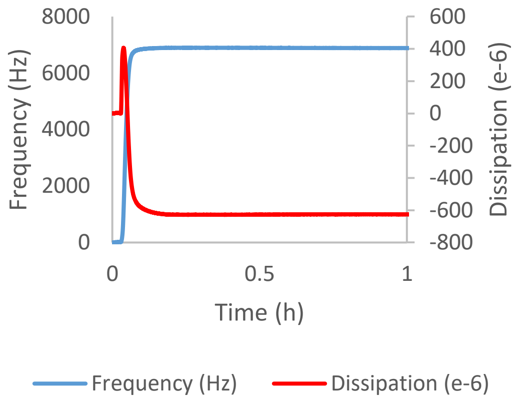
- 0.01% Triton X-100 – LS



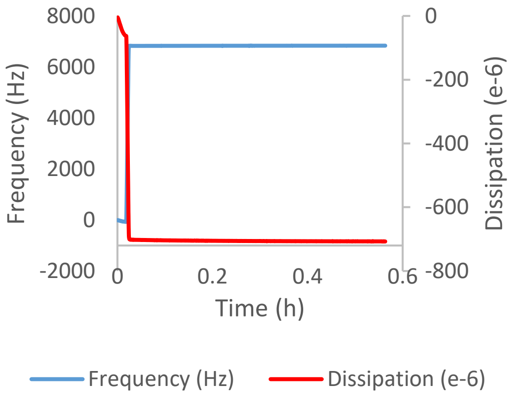


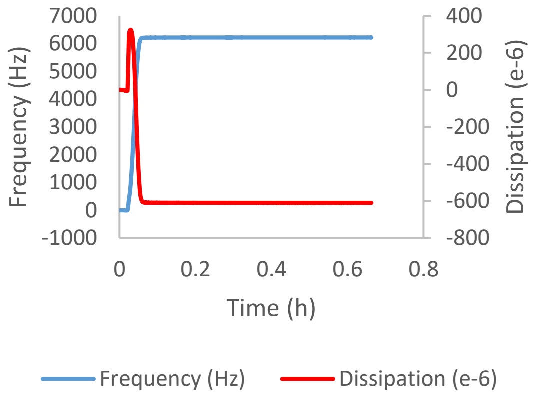
- 0.015% Triton X-100 – LS



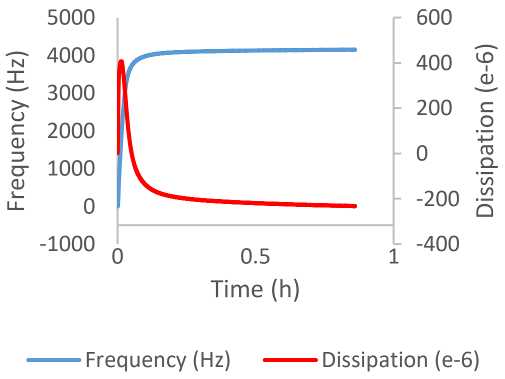


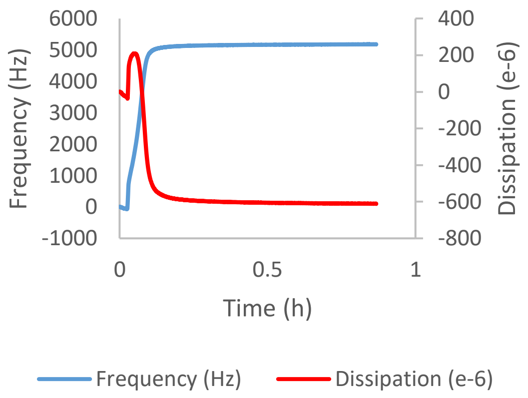
- 0.02% Triton X-100 – LS





- 0.001% Triton X-100 – MS





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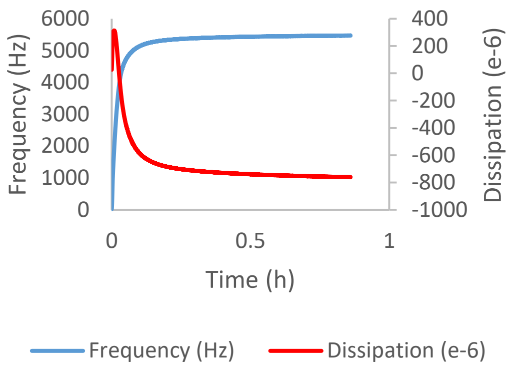
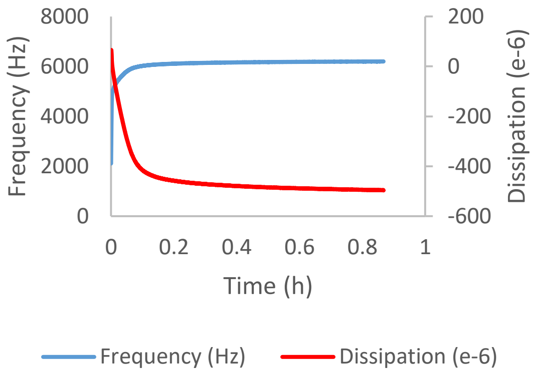
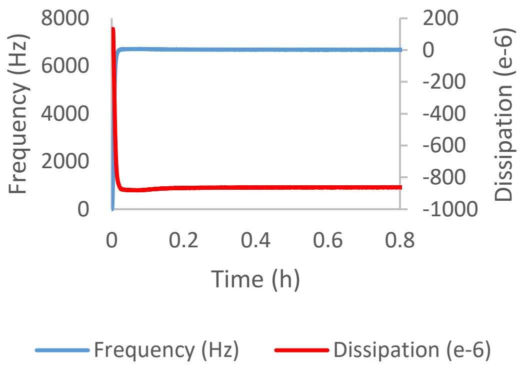
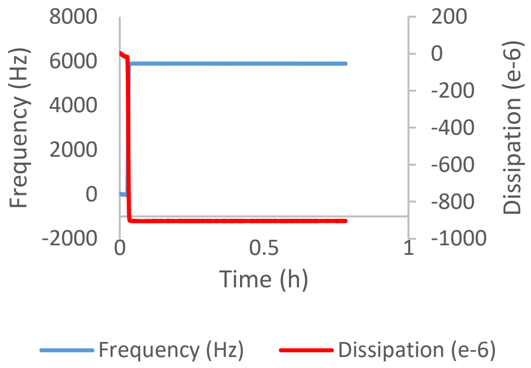


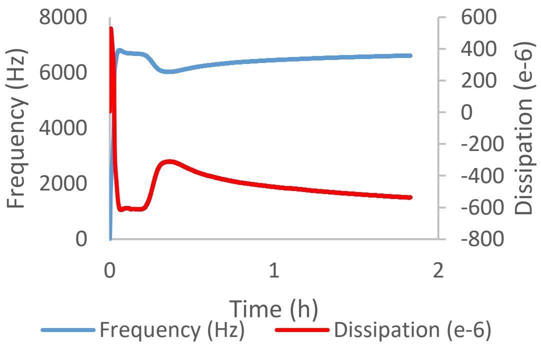


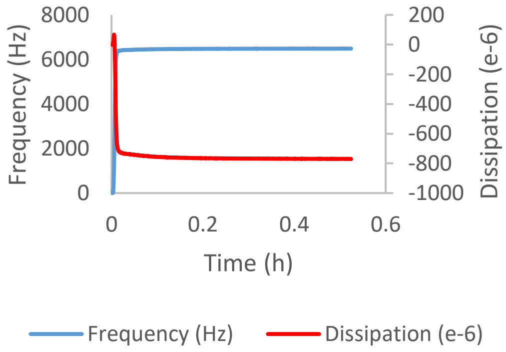
- 0.002% Triton X-100 – MS



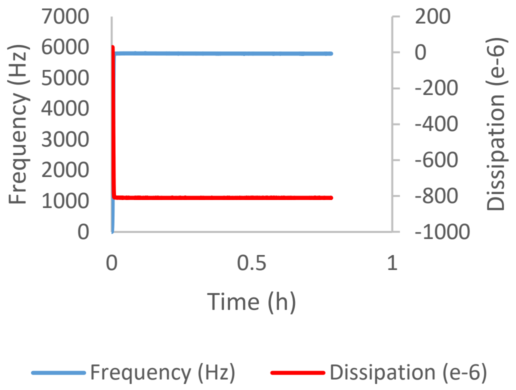


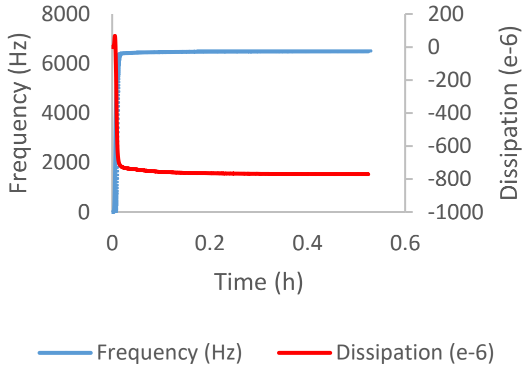
- 0.005% Triton X-100 – MS





- 0.01% Triton X-100 – MS





- 0.015% Triton X-100 – MS