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Wax Deposition as a Function of Flow Regimes

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Preface

TPG4920 - Petroleum Engineering, Master's Thesis is a compulsory subject in the Master of Science Petroleum Technology education with the specialization Petroleum Production at the Norwegian University of Science and Technology(NTNU). This work was carried out from January 15 to June 11 2018, and the topic was developed in collaboration with supervisor Harald Arne Asheim from the Department of Geoscience and Petroleum (IGP), and co-supervisors Ole Jørgen Nydal and Even Solbraa from the Department of Energy and Process Engineering (EPT).

I would like to thank Harald Arne Asheim for all help and guidance during this semester regarding this thesis. I would also like to thank Ole Jørgen Nydal and Even Solbraa for all discussions and guidance at the Multiphase Lab at EPT.

I would also like to thank co-supervisor Yury Novoseltsev for allowing me to use the wax deposition rig that he built at the Multiphase Lab. Almost all of the results in this master thesis come from experiments conducted at this wax deposition rig, and it would not have been possible to write this thesis without his help

Trondheim, June 11th 2018

Kristian Døble

Summary

As petroleum fluids flow in subsea pipelines, the fluids are cooled by the surrounding water. Many crude oils contain substantial amounts of wax molecules, and if the temperature of the fluids drops below the Wax Appearance Temperature(WAT), wax will precipitate and can deposit on the pipe wall. If the deposition is left untreated, the effective pipeline diameter will decrease, resulting in a reduction in flow capacity. In cases where the production is stopped without heating or insulation of the pipe, the wax deposit can completely plug the pipe.

Wax deposition during multiphase flow is not well understood, and only a few experimental studies has been conducted. 17 tests were therefore conducted using a multiphase flow rig at NTNU, where air and waxy oil were used as gas and liquid phase. Constant air rate was used, and different flow regimes were achieved by varying the oil rate. After each test, the wax deposition thickness and porosity were measured, and pictures were taken inside the copper pipe to determine the deposition pattern.

During stratified flow the wax deposited only on the bottom part of the pipe, and the wax thickness increased with increasing oil fraction, because more of the pipe was wetted.

Observations revealed that the thickest wax deposition was found in the transition region between stratified and slug flow, called wavy stratified flow. Here, the wax deposited in the bottom part of the pipe, but also on the top part of the pipe due to small droplets leaving the stratified flow, and instantaneously deposited when hitting the cold pipe wall.

During slug flow, the wax deposited around the whole pipe circumference, as the pipe wall was completely wetted. However, the wax thickness decreased with increasing oil rate due to the shear removal effect where already deposited material was mechanically removed by the flowing fluid.

Pictures taken inside the copper pipe after each test confirmed that some of the wax deposited due to gravity settling. This contradicts the common belief among researchers where deposition due to gravity settling is normally assumed negligible.

Sammendrag

Når olje og gass strømmer i undervannsrørledninger blir disse fluidene avkjølt av havvannet rundt røret. Mye av verdens råoljer inneholder betydeliger mengder med voks, og hvis temperaturen i røret faller under voksutfellesestemperaturen(WAT), så vil voksen i oljen utfelles og kan avsettes på rørveggen. Hvis avsetning ikke blir fjernet, så vil den effektive rørdiameteren minke, noe som resulterer i en reduksjon i strømningskapasiteten. I tilfeller der produksjonen stoppes uten oppvarming eller isolasjon av røret, kan voksavsetningen plugge igjen hele røret.

Voksavsetning i flerfasestrøm er ikke fullt ut forstått, og bare noen få eksperimentelle studier har blitt utført. 17 tester ble derfor utført ved bruk av en testrigg på NTNU, hvor luft og olje med oppløst voks ble brukt som gass- og væskefase. Konstant gassrate ble brukt, og forskjellige strømningsregimer ble oppnådd ved å variere oljeraten. Etter hver test ble voksavsetningens tykkelse og porøsitet målt, og bilder ble tatt inne i kobberrøret for å bestemme avsetningsmønsteret.

Under stratifisert strømning ble voksen kun avsatt på bunnen av røret, og tykkelsen økte etterhvert som oljefraksjonen i røret økte, fordi en større del av røret var fuktet.

Observasjoner viste at den tykkeste voksavsetningen ble funnet i overgangsregionen mellom stratifisert og slug strømning, ofte kalt bølgete stratifisert strømning. Her ble voksen avsatt både på bunnen men også på den øvre delen av røret på grunn av små dråper som ble løftet ut av den stratifiserte strømningen, og avsatt umiddelbart når de traff den kalde rørveggen.

Under slug strømning ble voksen avsatt rundt hele røromkretsen, siden rørveggen var totalt fuktet. Vokstykkelsen minket imidlertid med økende oljerate på grunn av skjærfjerningseffekten, der allerede avsatt materiale ble mekanisk fjernet av de strømmende fluidene.

Bilder tatt inne i kobberrøret etter hver test bekreftet at en del av voksen ble avsatt på grunn av tyngdekraftsmekanismen. Dette motsier den felles forståelsen blant forskere der avsetning på grunn av tyngdekraftsmekanismen normalt antas som ubetydelig.

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Nomenclature

Abbreviations

ASTM	American Society for Testing and Materials
CPM	Cross Polarization Microscopy
DSC	Differential Scanning Calorimetry
EPT	Department of Energy and Process Engineering
IGP	Department of Geoscience and Petroleum
NTNU	Norwegian University of Science and Technology
WAT	Wax Appearance Temperature

Roman Symbols

$\dot{m}$	Fluid Mass Flow	$[kg/s]$
$\frac{dC}{dr}$	Radial Concentration Gradient	$[m]$
$\frac{dP}{dL}_a$	Acceleration Pressure Drop	$[Pa]$
$\frac{dP}{dL}_f$	Frictional Pressure Drop	$[Pa]$
$\frac{dP}{dL}_g$	Gravitational Pressure Drop	$[Pa]$
D_B	Brownian Diffusion Coefficient	$[cm^2/s]$
D_D	Diffusion Coefficient	$[cm^2/s]$
d_w	Effective Pipe Diameter	$[m]$
m_{dep}	Mass of Deposited Wax	$[kg]$
N_{re}	Reynolds Number	

Q_g	Gas Flow Rate	$[m^3/s]$
Q_l	Liquid Flow Rate	$[m^3/s]$
A	Area over which deposition occurs	$[m]$
d	Pipe Diameter	$[m]$
f	Friction Factor	
L	Length of Pipe	$[m]$
r	Pipe Radius	$[m]$
t	Time	$[s]$
t,ref	Reference Time	$[s]$
v	Fluid Velocity	$[m/s]$

Greek Symbols

δ	Wax Thickness	$[mm]$
ϵ	Roughness	$[\mu m]$
μ	Viscosity	$[mm^2/s]$
ϕ	Wax Porosity	
ρ_o	Oil Density	$[kg/m^3]$
ρ_{dep}	Wax Deposition Density	$[kg/m^3]$
u_{sg}	Superficial Gas Velocity	$[m/s]$
u_{sl}	Superficial Liquid Velocity	$[m/s]$

Chapter 1

Introduction

Transportation of petroleum fluids from the wellhead to the receiving facilities is a vital part of petroleum production, and multiphase production pipes are increasingly used. Researchers have reported that approximately 85% of the world's crude oils encounter problems from wax formation, and wax deposition has been a flow assurance challenge for the petroleum industry since its inception(Thota and Onyeanuna, 2016). As the petroleum production frontiers even more extreme conditions, with deeper and colder water, the challenge is even more visible.

The increasingly longer multiphase pipelines located at the seabed transporting hydrocarbons from the wellhead to the platform or to onshore receiving facilities cools the flowing fluids. If the temperature of the fluid at any point in the pipe drops below the Wax Appearance Temperature(WAT), wax will precipitate as wax crystals. The wax does not necessary deposits on the pipe wall, often the wax crystals tend to disperse in the fluid and end up at the receiving facilities onshore. However, if the precipitated wax crystals do deposit on the pipe wall and is left untreated, the wax build-up can drastically decrease the efficiency of the transfer system, resulting in reduced production flow. In cases where the production stops without heating or insulation, the entire pipe can be blocked by the deposits.

Many experimental studies have been performed on wax precipitation and deposition in offshore pipelines. However, most of the studies are only conducted on single-phase flow. As multiphase flow is encountered in most of the pipelines in the world, many of those studies are not applicable in real situations. Multiphase flow is more complex than single-phase flow, due to the simultaneous presence of different phases, and different compounds in the same stream, making the understanding of deposition in multiphase flow more challenging. Only four experimental studies have been carried out to investigate this subject.

In this master thesis, wax deposition theory is described and explained, and later a

small scale multiphase rig at the Department of Energy and Process Engineering(EPT) is used to experimentally investigate the effect flow regimes and increased oil fraction has on the wax deposition. Which mechanisms responsible for the wax to deposit is also investigated. Oil containing 10wt% wax is circulated together with air through a cobber pipe cooled down by counter flowing cold water, and the wax deposition thickness and wax porosity are measured by direct and indirect methods. This work intends to identify the desirable flow regime to minimize wax deposition, and to decide if the wax thickness can be determined using The Pressure Drop Method. Which mechanisms that contributes to the deposition will be investigated by taking picture of the deposition pattern after every test.

Chapter 2

Theory

2.1 What is wax?

Petroleum crude oil is a mixture of different hydrocarbons, including paraffins, aromatics, asphaltenes and resins, and most of the crude oils in the world contain substantial amounts of wax molecules. An exact definition of wax has yet not been established, but wax usually refers to hydrocarbon components that are solid at ambient temperature and pressure but turns into liquid with higher temperatures. Venkatesan (2004) proposed the definition that waxes consists primarily of long chain, normal alkanes with chemical formula C_nH_{2n+2}, and carbon chain lengths from C_{20} to C_{78}. However, it is not only normal alkanes (sometimes called normal paraffins) that are denoted as waxes, but also cyclic or branched structures like iso-paraffins and cyclo-paraffins may also precipitate as waxes. Figure 2.1 shows examples of hydrocarbon components that forms waxes, such as normal alkanes, iso-paraffin and cyclo-paraffin

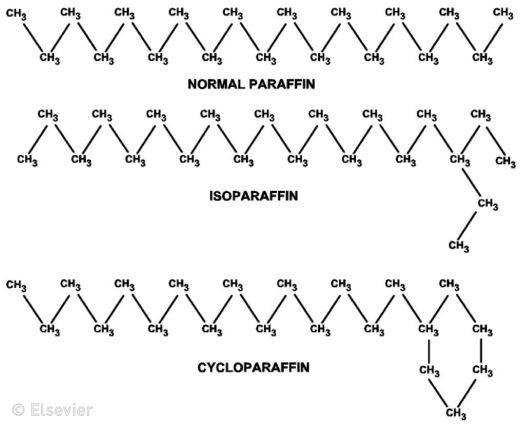
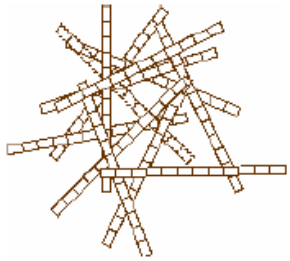
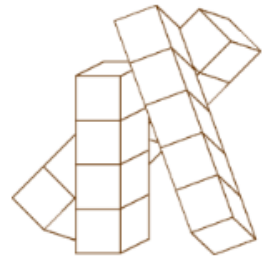


Figure 2.1: Examples of wax-forming components (Lira-Galeana and Hammami, 2000)



(a) Microcrystalline Wax Structure



(b) Macrocrystalline Wax Structure

Figure 2.2

Waxes are normally divided into two categories, namely microcrystalline and macrocrystalline waxes. The relative low molecular weight normal alkanes ($C_{20} - C_{40}$), are macrocrystalline waxes, and crystallizes as large needles and plates (Roenningsen et al., 1991). Macrocrystalline wax structure is shown in figure 2.2b, and a cross polarized microscopy image of macrocrystalline waxes is shown in figure 2.3. The white coloured parts in figure 2.3 are the wax forming platelet structures that interlocks, and the black parts consist of oil trapped inside the wax crystalline structure. This phenomenon of entrapped oil inside the wax structure will be discussed later. Macrocrystalline wax deposits in pipelines and causing flow assurance problems, especially in harsh environments as in long subsea pipelines, and will be the main focus in this thesis.

Microcrystalline wax are higher molecular weight normal alkanes or iso-alkanes, and the structure is shown in figure 2.2a. Microcrystalline waxes does not gives rise to as much flow assurance problems as macrocrystalline waxes, but it contributes to most to tank bottom sludges (Misra et al., 1995)
In this thesis, only the macrocrystalline waxes will be investigated, and further will be denoted just as waxes.

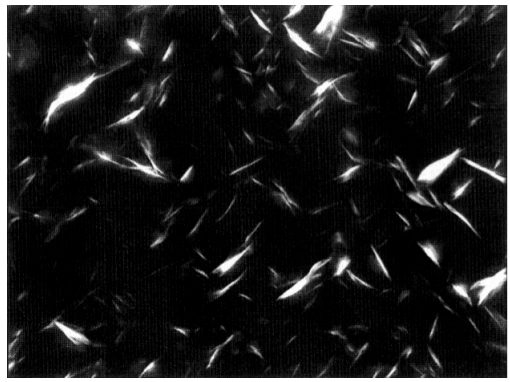


Figure 2.3: Cross Polarized Microscopy image of Wax oil gel Holder (1965)

2.2 Wax Appearance Temperature

One of the most important terms to describe and understand is the Wax Appearance Temperature (WAT). When the crude oil flowing in subsea pipelines cools, wax molecules starts to precipitate. WAT is defined as the temperature where the first wax molecule precipitates because the solubility limit is exceeded, and is interchangeably denoted as cloud point. When the temperature drops below WAT, the wax crystals start to form clusters of aligned chains, and if the temperature is held constant or further dropped, these clusters continue to grow and become stable. Once these clusters are stable they can interconnect with nearby cluster and grow larger. Formation of the crystals can change the appearance from transparent to cloudy, hence the name cloud point. Thermodynamically, WAT is defined as the maximum temperature at a given pressure where both the solid and liquid phase coexist in equilibrium (Towler et al., 2011). An exact measurement of WAT is very important in order to know when precipitation may start. Other synonyms of WAT are Wax Appearance Point, Wax Formation Temperature and Cloud Point. However, in this thesis the abbreviation WAT will further be used consequently.

2.2.1 Determination of WAT

Even though many techniques and measurement methods have been proposed to determine the WAT, not a single method gives a 100% accurate estimate. The problem lies in that none of the techniques manages to detect the appearance of the very first crystal. All the methods need a substantial amount of wax crystals to form before it is detectable. In addition, most petroleum fluids that contain wax molecules has a low precipitation rate just below WAT, making it even harder to detect the first crystals to appear.

Some of the most used methods for the determination of WAT are

- ASTM visual method
- Viscometry Method
- Differential Scanning Calorimetry (DSC)
- Cross Polarization Microscopy (CPM)

The simplest method is the American Society for Testing and Materials(ASTM) visual method, where a transparent jar containing 40ml of crude oil is heated and then cooled down in an isothermal bath. The temperature where the crude oils cloudiness is first visually observed is determined as the WAT. This method is expected to yield a WAT lower than the true WAT, because it is needed a substantial amount of wax crystal present for a naked eye to detect it.

Cross Polarization Microscopy(CPM) is generally accepted as the most sensitive method for the determination of WAT. Preheated samples of waxy oil are put into micro-capillaries, where the precipitated wax crystals will appear in a CPM as bright interference patterns in the polarized light. Even though the CPM technique yields good estimates of WAT, the method is very time demanding, and a skilled human interpreter is needed to use the

equipment.

The Differential Scanning Calorimetry(DSC) method uses the fact that wax precipitation is an exothermic process, and detects the heat emitted from crude oil in the phase transition from liquid to solid state. A sample is first heated to 80 degrees Celsius and then cooled down with a constant cooling rate, and the temperature when an exothermic change associated with precipitation of wax molecules is seen will be determined to be the WAT. (Jiang et al., 2001)

The Viscometry Method is based on the fact that the viscosity of solid state wax molecules is higher than in the liquid phase, thus changing the viscosity of the crude oil substantially. The viscosity of most crude oils without wax present is linearly increasing with decreasing temperatures. However, if wax is present in the oil, then a noticeable deviation should be seen when wax starts to precipitate. Typically, the viscosity is plotted against temperature, and the temperature where the curve starts to deviate from linearity is determined as WAT(Dantas Neto et al., 2010). Figure 2.4 shows an example of a plot of viscosity against temperature, and where the WAT would have been determined to be around 31 degrees Celsius

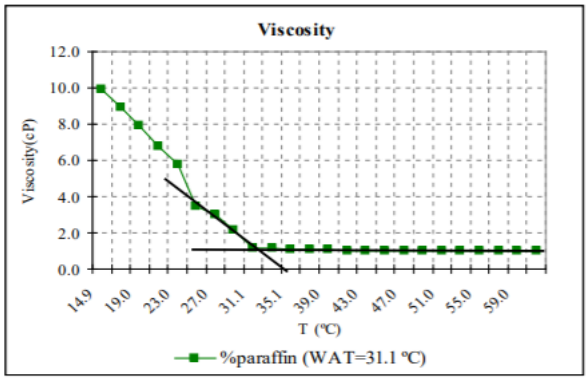


Figure 2.4: Determination of WAT by viscometry Dantas Neto et al. (2010)

Comparative studies have been performed to determine which method gives the best estimate for the true WAT value. Dantas Neto et al. (2010) used raw paraffin dissolved in turpentine, diesel, normal paraffin, naphtha-petrochemical, hexane, and LCO, and compared the WAT values obtained from viscosity and photoelectric signal method. He concluded that because the viscometry method needs a large amount of solid state wax crystals to detect the change in the viscosity, the WAT value was lower than the value obtained from the photoelectric signal method, where only a small amount is needed before is detectable.

Kok et al. (1996) studied the WAT from 15 different crude oils using DSC, thermomicroscopy and viscometry, and compared the results. He concluded that because of the great diversity of crude oils, it is impossible to state which of the methods that are most suitable for WAT determination.

Coutinho and Daridon (2005) concluded that the true WAT is not accessible experimentally by current available measurement techniques, and all the measurements should only be used as a guidance.

2.3 Removal of wax deposition

Removal methods of wax deposition can be classified into three categories, namely thermal, chemical and mechanical Removal Techniques

As wax precipitation is temperature dependant, thermal methods such as heating, or addition of hot water can be used to remove depositions. Heating is a very effective removal method, as the wax deposition will melt if it is raised above WAT. However, heating of long subsea pipelines is very costly, and a system for the addition of hot water must be in place.

Chemical additions such as solvents, surfactants or wax crystal modifiers may be added to remove wax deposition in production lines. Wax crystal modifiers are very effective, as they work on a molecular level, thus is more effective than solvents or surfactants that must be applied in large volumes.

Mechanical removal by pigging has been the preferred method for removal of wax depositions by the oil companies. The method allows the deposition to build up, before it is scraped off using a pigging tool, shown in figure 2.5. Normally, pigging is used on a frequent basis. However, the use of pigging has some risks accompanied with it, as the pig can get stuck if used incorrectly.

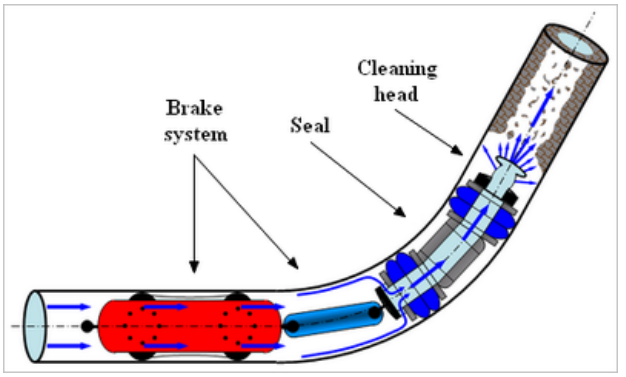


Figure 2.5: Pigging tool for wax removal

2.4 Wax Porosity

The wax deposited on the pipe wall is a gel of mixed wax components with oil trapped inside (Singh et al., 1999). The volumetric fraction of oil to the total deposition is called wax porosity, and is affected by the flow regime, oil composition, wall roughness etc. Burger et al. (1981) determined that wax deposited on the pipe wall formed a crystalline porosity structure shown in figure 2.6, with oil trapped in the structure .

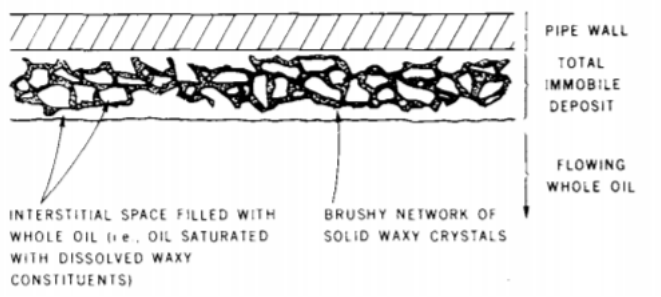


Figure 2.6: Network of solid wax deposited on the pipe wall, with space filled with oil

Wax porosity is important when deciding which method to use for wax removal. A higher oil content in the wax, meaning a higher wax porosity, implies a softer and easier removed deposition. Hard depositions are more difficult to remove, and there are examples of pipelines that had to be abandoned because the pig got stuck due to underestimating of the wax deposition and porosity. Therefore, to determine pigging frequency, the wax porosity is of great importance. Wax porosity value scan vary significantly, thus making it even more important to estimate correctly. Several wax porosity experiments have been conducted, and it was found out that for a soft wax layer the wax porosity could be as high as 90%, and 50-72% for hard depositions Lund (1998). The wax porosity was determined to be 80-86% by Burger et al. (1981), and Rygg et al. (1998) found the wax porosity for a gas oil system to be 60% .

It is shown that the wax porosity is also dependant on the cooling rate. Wax deposited rapidly due to a very cold pipe wall is expected to be softer than more slowly deposited wax. The rapid deposition rates traps the oil molecules inside the porosity structure of the depositions.

2.5 Wax Deposition Mechanism

Svendsen (1993) found out that three conditions must be fulfilled for wax deposition to occur. Logically, the temperature of the pipe wall must be lower than the WAT for the crystallization process to start. When the oil and gas enter the subsea pipeline from the reservoir it is cooled gradually along the pipeline. Heat transfers from the petroleum liq-

uids, through the pipe wall and into the cold surrounding water. Depending on the amount of insulation and heating of the pipe, the temperature can drop below WAT, thus allowing for wax crystals to form on the pipe wall.

The second condition is a negative radial temperature gradient must be present in the flow. The warm petroleum flowing in the bulk of the pipe and the colder pipe wall forms a radial temperature gradient. This radial temperature gradient is vital for wax precipitation, and zero temperature gradient implies that no wax deposition will occur. Usually, the subsea pipeline rests on the seabed or is covered under soil. If it is covered, the soil will insulate the pipe and allowing for a higher pipe wall temperature. The wax itself can also act like an insulation layer, reducing the temperature gradient from the pipe wall to the bulk of the fluid, thus limiting further deposition. Thicker deposition layer is usually seen further away from the wellhead, as there is also formed an axial temperature gradient due to the gradual cooling along the pipeline.

Wax precipitation does not necessarily end in deposition on the pipe wall. The third condition is that high enough wall roughness is needed for the wax crystals to deposit. Wax crystals tend to disperse in the fluid rather than settling on the pipe wall. A sufficient amount of wax crystals has to agglomerate with materials such as clay or asphaltenes to form larger crystals, and high enough wall roughness is needed for the wax crystals to stick and deposit on the pipe wall. Therefore, pipelines with lower wall roughness should always be chosen in production system known to have oil with wax components.

Several experimental studies have been performed to raise the understanding of deposition mechanism in single-phase flow, but they are not yet fully understood. Burger et al. (1981) identified four possible mechanisms for deposition of wax crystals to occur

- Diffusion
- Brownian Diffusion
- Gravity Settling
- Shear Dispersion

Bern et al. (1980) estimated the extent of wax deposition from crude oil lines, and the results indicated that the predominantly mechanism for deposition was diffusion. It is generally accepted that diffusion is the main deposition mechanism.

2.5.1 Diffusion

Diffusion is driven by the wax concentration gradient evolving when temperature at the pipe wall drops below WAT, leading to precipitation to occur first at the pipe wall. A thin laminar sub-layer with a radial temperature gradient is formed close to the wall. When the oil at the pipe wall drops below WAT, the oil is fully saturated, and wax starts to precipitate. Because of the temperature gradient rising radially towards the centre, the bulk

temperature is normally above WAT, and no wax is precipitated there. Since the solubility of wax is a decreasing function of temperature, this unevenness forms a wax concentration gradient between the bulk and the wall, and the wax molecules diffuse towards the wall. Once these molecules reach the interface between solid and liquid state, they will precipitate and be added to the solid deposition.

Fick's Law estimates the rate of diffusion, and is shown in equation 2.1

$$\frac{d_{m_{dep}}}{d_t} = -\rho_{dep} D_D A \frac{d_C}{d_r} \tag{2.1}$$

where m_{dep} is the mass of the deposition, ρ_{dep} is the deposition density, D_D is the diffusion coefficient of liquid wax in oil, A is the area over which deposition occurs, and $\frac{dC}{dr}$ is the radial concentration gradient

Diffusion is illustrated in figure 2.7

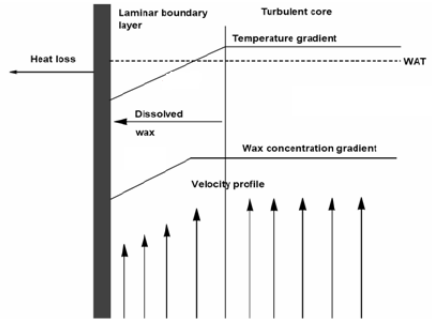


Figure 2.7: Wax Deposition by Diffusion (Shunsuke Hashimoto and Ohgaki, 2010)

2.5.2 Gravity Settling

Deposition due to the density difference from wax to the surrounding liquid, where wax is normally the heaviest one, is called gravity settling. Most researches assume gravity settling as insignificant in contributing to wax deposition. Burger et al. (1981) conducted experiments in both vertical and horizontal pipes, where the resulting deposition pattern were similar, thus concluding that deposition due to gravity settling is negligible. Singh et al. (2000) also conducted similar experiments and concluded likewise. However, gravity settling is not fully understood, and these experiments were conducted with high liquid velocities, so in multiphase cases or in cases with low liquid velocity gravity settling can play a role.

2.5.3 Brownian Diffusion

Small random movements of the wax crystals due to the collision between the crystals and thermally agitated oil molecules, are called Brownian movements. As there exist a wax concentration gradient as discussed in the Diffusion chapter, these Brownian movements transports the wax crystals to the pipe wall. The mathematical description of Brownian diffusion is the same as for diffusion, and is shown in equation 2.2

$$\frac{d_{m_B}}{d_t} = -\rho_d D_B A \frac{d_C}{d_r} \tag{2.2}$$

where m_B is the mass of the deposition due to brownian diffusion, and D_B is the Brownian diffusion coefficient.

Deposition due to Brownian diffusion has been considered as negligible by several authors such Brown et al. (1993) and Burger et al. (1981)

2.5.4 Shear dispersion

Most studies show that diffusion is the dominant deposition mechanism, but there are studies showing the impact of shear dispersion. Shear dispersion can act as both a deposition mechanism, but also a mechanism for removing of already deposited materials. When wax crystals are precipitated and dispersed in the fluid, they are usually dragged in the direction and with same speed as the flow. In many cases, the wax molecules will be dispersed in the fluid all the way into the receiving facilities such as separators. This situation is desirable, as it is much easier to remove wax deposition from a separator on a platform than from pipelines located at the seabed.

However, in the subsea pipeline the flowing crude oil causes shear forces, inducing lateral movements in the fluid. This lateral movement of the flow can transport wax molecules from the turbulent core to the boundary layer at the colder pipe wall where they will deposit. Deposition due to shear dispersion will only occur if there is a significant amount of precipitated wax in the bulk, and this will only happen if the temperature is close to, or even under the WAT. If the temperature in the bulk is high enough, no wax

will be precipitated and dispersed in the fluid, thus shear dispersion will not increase the total deposition.

Experiments have been conducted to investigate the effect of shear rate as a deposition mechanism. Assuming gravity settling and Brownian diffusion are negligible, the idea was to check if additional wax will be deposited under conditions where diffusion does not contribute, as with no radial temperature gradient. Therefore, two experiments were carried out under this condition of no radial temperature gradient, one at the University of Tulsa and the other at Porsgrunn flow loop (Gjermundsen, 2006). Both studies concluded that no deposition occurred, concluding that shear dispersion is an insignificant mechanism of deposition.

Shear dispersion can also remove already deposited materials, and this mechanism is often called shear removal, or sloughing. An increased share force can encourage deposition by lateral movements, but the same shear force increase can mechanically strip of earlier deposited wax (R. Bott and Gudmundsson, 1977). Shear removal occurs when the shear stress exerted by the fluid flow at the deposition interface is large enough to mechanically remove some of the depositions. Agrawal et al. (1990) found out that turbulence is the main factor for the decrease in deposition in turbulent single-phase flow, while diffusion is the main factor for the increased deposition with increasing flow rate in laminar single-phase flow.

Hsu et al. (1994) concluded similarly where a wax deposition system for turbulent flow was developed, and experiments showed that the sloughing effects generated under turbulent flow conditions has significant impact on wax deposition rate, and that the it cannot be neglected. The conclusion is that wax deposition can be reduced if the flow rate is increased during turbulent flow.

2.5.5 Aging

Singh et al. (1999) suggested that wax formation can be described as a combination of the following five steps

1. Formation of a wax gel layer on the surface of the cold pipe wall due to precipitation close to the pipe wall
2. The radial concentration gradient forms a mass flux of dissolved waxes towards the wax gel layer
3. Internal diffusion of some of these wax molecules inside the gel
4. Precipitation of these wax molecules within the gel deposit
5. Counter-diffusion of de-waxed oil out of the wax gel deposit

The first two steps have already been discussed, explaining how diffusion increases the deposition layer. The three next steps explain a phenomenon called aging, sometimes

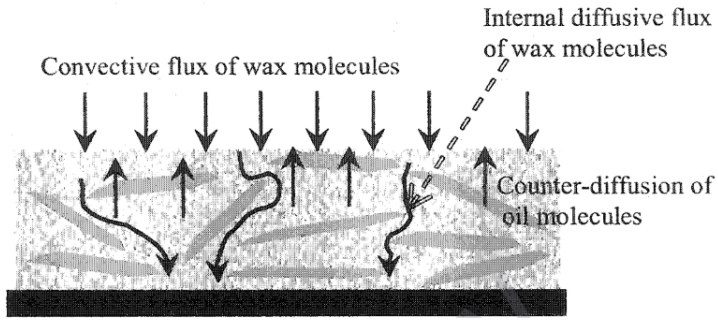


Figure 2.8: Graphical representation of the wax deposition and aging process

written ageing. The aging effect is related to the shear removal effect, and figure 2.8 shows a graphically representation of wax deposition and aging

After the first wax is deposited on the pipe wall, new wax molecules are transported to the pipe wall through diffusion. The wax molecules in the deposition are not immobile, but they diffuse internally in the deposition towards the pipe wall. At the same time, counter diffusion of oil trapped inside the wax crystal structure occurs. This is possible due to the gel acting like a porous medium where wax and oil can move inside. This counter diffusion allows some of the oil to escape into the bulk, thus leaving the remaining wax deposition with lower wax porosity, i.e. the deposition is getting harder over time. This process of increasingly harder deposition is dependant of the oil rate.

Singh et al. (2000) conducted several experiments using oil solvent and food grade wax to investigate the effects of increased oil rate and concluded that an increase in the oil rate lead to a decrease in both the thickness and the wax porosity of the deposition. The increased oil rate encourages more oil to counter diffuse out of the wax gel layer, leaving the remaining deposition harder. The increased oil rate also decreased the total wax thickness due to the increased shear rate.

Zhang et al. (2002) showed that the aging phenomenon affected the wax porosity by hardening the deposited, but the total build-up of deposition was unaffected. In other words, the volumetric amount of trapped oil that counter diffused out of the crystal structure, was replaced with a same amount of wax, leaving the wax thickness independent of the aging process.

Brown et al. (1993) conducted several experiments on wax porosity for different multiphase flow regimes and discovered that flows with high shear rates resulted in harder deposits than the deposits formed at low shear rates. A flow regime with high shear force such as slug flow leads to a smaller wax porosity than a low shear force regime such as stratified flow.

2.6 Multiphase Flow

Until now, only single-phase flow has been discussed. However, for the understanding of wax deposition during multiphase flow, some concepts must be defined. Multiphase flow is usually defined as a flow of two or more materials with different states(gas, liquid, solid) or with similar states but different chemical properties. In the petroleum industry, examples of multiphase flow are oil-gas two phase flow, or oil-gas-water three phase flow.

When two or more substances flow inside a pipe, different geometrical configurations called flow regimes occurs. single-phase flow is normally divided into two regimes, laminar and turbulent flow. Multiphase flow regimes are more complex due to the large varieties of velocities and composition of the different components. In addition, flow regimes in a vertical pipe is not similar as in a horizontal pipe. Flow regimes in horizontal pipes are usually more complex than in vertical pipes because of the gravity force. In a horizontal pipe the gravity acts perpendicularly to the flow direction, forming a density segregated flow in the pipe if the fluid velocities are low enough. This axisymmetric flow is important to understand. If the fluid velocities are large, flow regimes that are not segregated by density can arise, such as slug or bubble flow.

Multiphase horizontal flow regimes are normally divided into five different patterns. The different flow regimes are show in figure 2.9 and are listed under

- Stratified Flow
- Wavy Stratified Flow
- Slug Flow
- Annular Flow
- Dispersed Bubble Flow

Determination of the dominant flow regime can be challenging, as the flow regime is not always well defined. The flow pattern is influenced by the inclination and diameter of the pipe, and the physical properties of the fluids and their flow rates. Especially in the transition regions the determination can be challenging, and visual observation of the multiphase flow is often used to decide which flow regime that is dominant.

There also exist methods for calculation of which flow regime most likely to occur. Zhang et al. (2002) developed a unified model for the prediction of flow regime transitions in horizontal pipe lines. The transition between stratified and slug flow was predicted by solving the momentum equations, and validation of this model has proven good results.

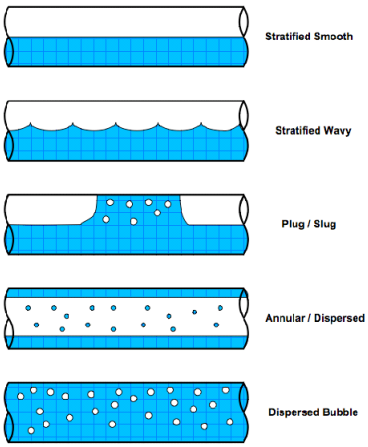


Figure 2.9: Horizontal two phase flow regimes

Superficial velocities for oil and gas can be used to draw flow regime maps. The superficial velocity is an artificial velocity defined as the speed a given phase would have if it was occupying the whole cross-sectional area for itself. The superficial velocity is not a physically real velocity, but is a very convention parameter for analysis of different flow regimes, and is mathematically expressed in equations 2.3 and 2.4

$$u_{sl} = \frac{Q_l}{A} \tag{2.3}$$

$$u_{sg} = \frac{Q_g}{A} \tag{2.4}$$

where u_{sg} and u_{sl} are the gas superficial velocity and liquid superficial velocities(m/s), Q is the flow(m^3/s) for that phase, and A is the cross-section area of the flow(m^2).

Another important parameter is the holdup factor. Holdup is is illustrated in figure 2.10, and is defined as the fraction of a specific fluid present in an interval of pipe, and the mathematical expression is shown in equation 2.5

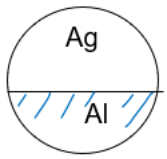


Figure 2.10: Graphical representation of holdup

$$H_l = \frac{A_l}{A} \tag{2.5}$$

where H_l is the liquid holdup, A_l is the cross-sectional area occupied by the liquid, and A is the total cross-sectional area.

In a two-phase gas/liquid flow, the area not occupied by the liquid will of course be occupied by the gas, giving the following equation

$$H_g = 1 - H_l \tag{2.6}$$

where H_g is the gas holdup.

The gravitational force acting on the multiphase flow forces the fluids to flow at different speeds, normally the heavier a fluid the slower it flows, or say being more held up. This difference in fluid velocities makes the volumetric fraction of the oil at any point in the pipeline to be greater than the input volume fraction of the pipe. Holdup factor must not be confused with the cut, which is the total flow rate due to that fluid.

The liquid holdup is important in the prediction of wax deposition, as a surface not wetted by the liquid phase will not be exposed to wax deposition. The liquid holdup can be measured both directly and indirectly. When using the direct method, quick acting closing valves are needed to suddenly isolate the pipe section and trap the fluids inside. Afterwards, the trapped fluids are measured, and thus the holdup can easily be calculated when knowing the pipe geometries. This method yields results based on direct measurements, however it has some uncertainties. The quick acting closing valves must trap the exact amount of gas and liquid present at the closing time, and if the volume of the pipe section is low, small errors in measurement can give large errors in the results.

To predict the liquid holdup of a flow indirectly, one must know the flow regime for that given pressure and temperature, superficial velocities etc. Even though several literature models have been developed for the prediction of liquid hold up indirectly, they are not accurate and there is no clear understanding which model to use for the different situations. The most famous model is the model for prediction of liquid holdup in stratified flow developed by Taitel and Dukler (1976), and most later studies are based upon this paper.

2.7 Wax Thickness Measurements

Many methods have been proposed to determine the wax deposition thickness, and four of these methods are

1. Spool Piece Method
2. Pigging Method
3. Pressure Drop Method
4. Heat Transfer Method

2.7.1 Direct Measurement Methods

The Spool Piece and Pigging Method are direct methods, whereas The Pressure Drop and Heat Transfer Methods are indirect methods. The Spool Piece Method is based on the possibility to remove the test section after each run, and the wax thickness is determined by measuring the weight and the volume of the wax. When the weight and volume of a clean and empty spool piece is known, back calculations are easily made. This method yields results based on direct observations and measurements and is widely used in experimental deposition studies with low pressure. If this method is used in high pressure systems, the depressurizing when dismantling spool piece can change the phase equilibrium. Hence, this method yields best results when used in low pressure systems. However, the method is time consuming, as the test section has to be bypassed or even totally dismantled after each run. This also induces some uncertainties, as the repeatedly dismantling of the test section can lead to small changes in pipe and angle dimensions.

When the weight of the wax deposition is known, the wax thickness is calculated in equation 2.7

$$\delta = r - \sqrt{r^2 - \frac{m_{dep}}{\rho_{dep}\pi L}} \tag{2.7}$$

where r is the inner radius of the empty pipe, m_{dep} is the weight of the deposited wax, ρ_{dep} is the density of the wax, and L is the length of the spool piece.

As discussed previously, the wax deposition is a mixture of wax with trapped oil inside. As both the density of oil and pure wax is known, the density of the deposition is calculated in equation 2.8

$$\rho_{dep} = \rho_{oil}(\phi) + \rho_{wax}(1 - \phi) \tag{2.8}$$

where ϕ is the wax porosity.

When using The Spool Piece Method, the calculated wax thickness is an average thickness across the test section. The actual thickness at a given point cannot be determined using this method, however the average thickness is assumed to be a good representation

of the amount of wax deposited.

For The Pigging Method, a spherical pig is run through the spool piece removing all the deposition. Measurements such as weight and volume are conducted on the removed deposition. Pigging is widely used in the field; thus, the technique is well known. However, for laboratory studies, The Spool Piece Method is more used.

2.7.2 Indirect Measurement Methods

The Pressure Drop and Heat Transfer Method use measurements of pressure and heat during experiments to determine wax thickness. The Heat Transfer Model is based on the idea that the wax deposition on the pipe wall forms an extra thermal resistance due to heat conduction through the wax layer, that adds to the total thermal resistance from the flowing fluid to the environment. This extra thermal resistance is approximately in direct proportion to the thickness of the wax layer. Even though this method has shown promising results, this method will not be investigated further in this thesis.

The Pressure Drop Method is based on the fact that the effective pipe diameter of the pipe is reduced due to the deposition along the pipe wall, inducing additional friction pressure drop. The Pressure Drop Method is developed for single-phase flow, and no indirect method for the determination of wax thickness in multiphase flow has been developed. However, attempts can be made to use The Pressure Drop Method to determine the wax deposition in multiphase flow.

Figure 2.11 shows a cross section of a pipe segment with relevant variables for determination of wax thickness

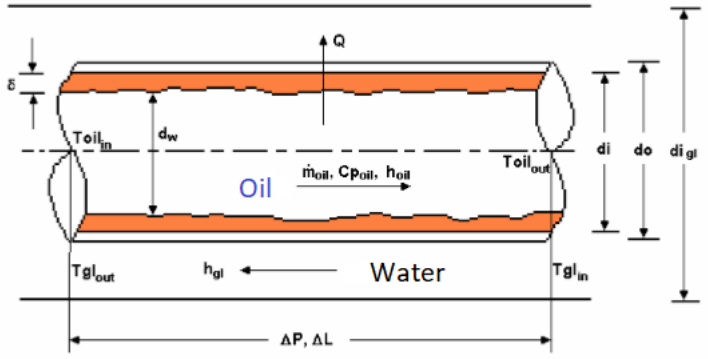


Figure 2.11: Cross section of Pipe Segment and Relevant Variables

By applying momentum balance, the pressure gradient in a pipe segment for steady state conditions is calculated in equation 2.9

$$\frac{dP}{dL} = \frac{dp}{dL}_f + \frac{dP}{dL}_g + \frac{dP}{dL}_a cc = -\tau\frac{\pi d}{A} - \rho g sin(\theta) - \rho v\frac{dv}{dL} \tag{2.9}$$

The total pressure drop is equal to the sum of the gravitational, frictional and acceleration pressure drop. As the test section is horizontally oriented, the gravitational pressure drop is effectively zero. In addition, the acceleration pressure drop can be neglected for incompressible flow, and air is normally considered incompressible for low velocities. This gives the following equation

$$\frac{dP}{dL} = \frac{dP}{dL}_f = -\frac{f\rho v^2}{2d_w} \tag{2.10}$$

where f is the friction factor, ρ is the density of the fluid, v is the velocity, and d_w is the effective diameter of the pipe.

The ratio between pressure drop at time, t, and at a reference time, ref, is expressed in equation 2.11

$$\frac{\frac{dP}{dL}t}{\frac{dP}{dL}ref} = \frac{-\frac{f_t\rho_t v_t^2}{2d_{w,t}}}{-\frac{f_{ref}\rho_{ref}v_{ref}^2}{2d_{w,ref}}} \tag{2.11}$$

At reference time, the thickness is assumed to be zero. Equation 2.11 can then be expressed as in equation 2.12

$$\delta = \frac{d_i}{2}\left(1 - \left(\frac{f_t\rho_t\dot{m}_t^2}{f_{ref}\rho_{ref}\dot{m}_{ref}^2}\frac{\frac{dP}{dL}ref}{\frac{dp}{dL}t}\right)^{\frac{1}{5}}\right) \tag{2.12}$$

where δ is the thickness of the wax deposition, and $\dot{m}$ is the mass flow of the fluid. This equation can be used in both laminar and turbulent flow.

For laminar flow, the Moody friction factor is given in equation 2.13

$$f = \frac{64}{N_{re}} \tag{2.13}$$

where N_{re} is the Reynolds number. Reynolds number is given in equation 2.14

$$N_{re} = \frac{\rho v d_h}{\mu} \tag{2.14}$$

where d_h is the hydraulic diameter of the pipe, and μ is the dynamic viscosity of the fluid.

The Moody friction factor for turbulent flow can be determined using the Haaland equation

$$\frac{1}{\sqrt{f}} = -1.8 \log \left[\left(\frac{\epsilon/D}{3.7} \right)^{1.11} + \frac{6.9}{N_{re}} \right] \tag{2.15}$$

The pressure drop in single-phase air flow can be measured before and after the wax deposition. Since both the density and the mass flow are equal before and after wax deposition, 2.12 can be written like this

$$\delta = \frac{d_i}{2} \left(1 - \left(\frac{f_t}{f_{ref}} \frac{\frac{dP}{dL}_{ref}}{\frac{dp}{dL}_t} \right)^{\frac{1}{5}} \right) \tag{2.16}$$

The friction factors before and after wax deposition are assumed similar, giving following equation

$$\delta = \frac{d_i}{2} \left(1 - \left(\frac{\frac{dP}{dL}_{ref}}{\frac{dp}{dL}_t} \right)^{\frac{1}{5}} \right) \tag{2.17}$$

As mentioned before, the Pressure Drop Method is developed for single-phase flow. To determine wax thickness indirectly in multiphase flow is a more challenging task. A joint project between the US department of Energy, the University of Tulsa, and 23 private companies investigated the problem of wax accumulation in deep-water pipelines and assumed that neither The Pressure Drop nor The Heat Transfer Method were applicable in multiphase flow situation due to the increased inaccuracy of predicting multiphase flow pressure gradients and heat transfer (Sarica and Volk, 2004). However, as this is the only experimental study published on the use of The Pressure Drop Method in multiphase flow, further studies have to be conducted in order to make a conclusion.

2.8 Uncertainty

To calculate the percentage error of the results, the equation 2.18 was used

$$PercentageError = \left| \frac{value_{error} - value_{actual}}{value_{actual}} \right| \tag{2.18}$$

For cases where two different percentage errors were achieved, one for the case where the uncertainty value is added to the experimental value, and one where the uncertainty value is subtracted, the average percentage error was found by taking the average of the two values.

Results are assumed reliable if the percentage error is less than 5%. In cases where the percentage error is greater than 5%, the results should not be further considered.

2.9 Multiphase deposition Studies and Literature Review

Until now, only deposition during single-phase flow has been discussed. This is because most wax deposition studies focus on single-phase flow, and it is important to understand the more basic single-phase deposition before starting with the more complex multiphase deposition. Most production situations met in the petroleum industry are multiphase, making the single-phase studies not so relevant. Wax precipitation and deposition in multiphase flow is an area not well understood and is a at a preliminary research stage. The additional gas phase makes the calculations and deposition mechanisms more complex. Normally, multiphase wax deposition can be divided into three categories:

- Oil/gas two phase flow
- Oil/water two phase flow
- Oil/Water/gas three phase flow

For the oil/water wax deposition, some experiments and studies have been conducted (G.H. Couto et al, 2006), but will not be further investigated in this thesis.

For the oil/water/gas case, no experimental studies have yet been published due to its extreme complexity.

For the oil/gas wax deposition, there are limited published literature, and only four experimental studies have been performed until now (Gong et al., 2011), (Matzain et al., 2002a), (Kilincer, 2003) and (Sarica and Volk, 2004).

The deposition pattern is expected to be flow regime dependant. In stratified flow with low u_{sl}, the liquid phase will only be in contact with the pipe along the bottom part of the pipe, and the deposition will only occur there. Stratified flow is normally seen with u_{sl}, implying low shear forces. As discussed before, the shear forces make the wax molecules to diffuse out of the wax deposition, thus leaving the deposition harder and with a lower wax porosity. Lower shear force as seen in stratified flow will leave a soft deposit along the bottom part of the pipe. This was confirmed by Matzain et al. (2002a) where he used crude oil from the Gulf of Mexico with 6,6% wax content and natural gas from Oklahoma Natural Gas Company to determine wax thickness and deposition pattern for different flow regimes.

For slug flow, it is expected that deposition of wax will occur all around the circumference of the pipe due to the slug flow wetting the entire pipe. The shear forces induced by the slugs passing leave the deposition significantly harder than in stratified flow (Matzain et al., 2002b). The deposition distribution along the pipe circumference in a horizontal pipe made by Matzain et al. (2002b) is shown in figure 2.12

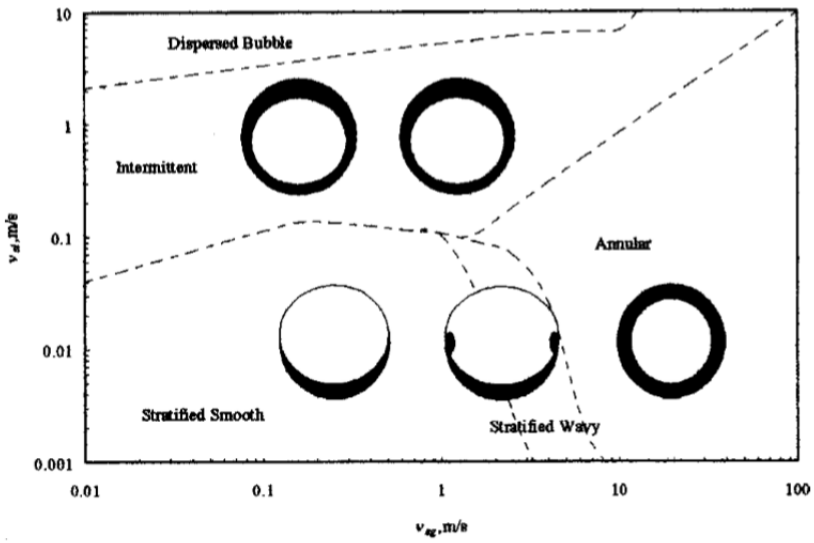


Figure 2.12: Wax thickness distribution for various horizontal flow patterns

Flow maps can be made to show which flow regime is expected for every u_{sl} and u_{sg}. However, these flow maps are just suggestive, not an exact science, and small changes in parameters can affect the dominant flow regime. Therefore, most studies have been performed in the definite regions rather than the transition regions.

Both Matzain et al. (2002a) and Kilincer (2003) investigated the effect different flow regimes have on the hardness of the deposition. The two experimental tests are not totally comparable as the liquid phases used were not similar. However, the qualitative studies made by these authors showed that the deposition in horizontal stratified flow gave softer and thicker deposition than in slug flow.

Chapter 3

Experimental setup and Methodology

Few test rigs have been built to investigate wax deposition patterns in multiphase flow. To build a large-scale test rig with reservoir conditions is expensive and challenging, so the solution was to build a small scale test rig to investigate the wax deposition with lower temperature and pressure conditions.

3.1 Rig at NTNU

A small scale test rig has been built by Yury Novoseltsev at the Department of Energy and Process Engineering (EPT) at NTNU, for the investigation of wax deposition in multiphase flow. The test section is a 2 meter long copper pipe with inner diameter 25 millimetre where waxy oil is circulated. Cold water at 5 degrees Celsius is run counter current in the annular between the cobber pipe and a transparent acrylic pipe to force cooling of the cobber pipe. Even though subsea pipelines situated on the seabed does not have flowing water along its side, the enormous amount of cold water is easiest simulated in the lab with flowing water cooled by a chiller. The test section with the cobber pipe situated inside the acrylic pipe is put horizontally on a table to ensure stable conditions and reliable data acquisition, and the setup is shown in figure 3.1

A heating tank is used to heat the waxy oil to 60 degrees Celsius, and K-type thermocouples are installed in contact with the oil at the outlet to ensures proper surveillance of the temperature. The loop is closed, where a Parker Petrol Pump, showed in figure 3.2 is used to pump the oil and wax mixture from the heating tank and into the cobber pipe in the test section through insulated flexible tubing, and back into the heating tank.

Air is taken from the central circulation system and brought into the system through a choke. The choke can manually be opened and closed to adjust the air flow into the

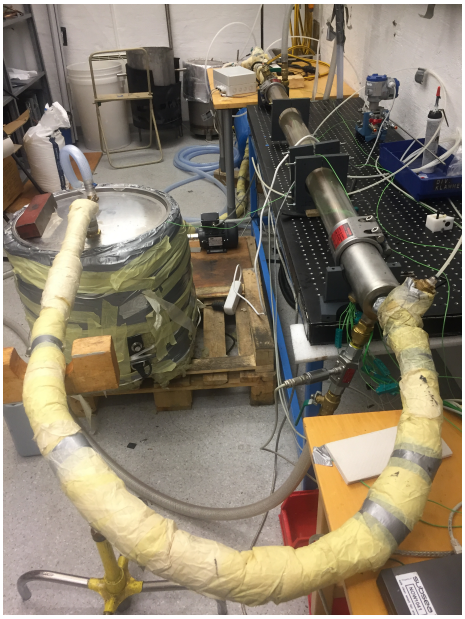
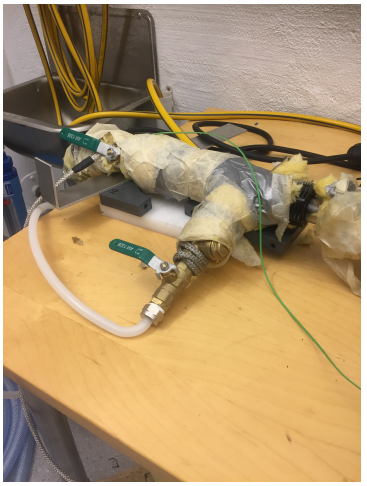


Figure 3.1: Overview of the wax rig

Figure 3.2: Parker Petrol Pump, used to pump waxy oil through the test section

system, and a Techfluid Glasstube Flow Meter $25 - 250l/min$ is used to measure the air flow rate. The mixing point of the oil and air is located 0.5 meter from the test section, and the mixture flows through a transparent acrylic pipe before entering the cobber pipe allowing for visual observations of the flow regime. The flow meter and the mixing point are shown in figure 3.3a and 3.3b

(a) Pressure Tank and Techfluid Flow Meter



(b) Mixing point of air and waxy oil

Figure 3.3

Heating tape is used at the inlet side of the test section, to ensure constant temperature of the waxy oil at the inlet side of the test section, and a Proportional–integral–derivative(PID) controller shown in figure 3.4, is used to control the heating tapes. Sometimes the heating in the heating tank is not sufficient to ensure a constant temperature, so the heating tapes provides additional heating. The temperature on the topside and bottom side along the pipe is measured by six K-type thermocouples that are placed on the pipe, three on top and three on the bottom. The thermocouples are connected to a computer, and the temperatures are logged using LabView. In addition, a Differential Pressure Cell with a range of 1000 pascal is used to measure the pressure drop over the test section

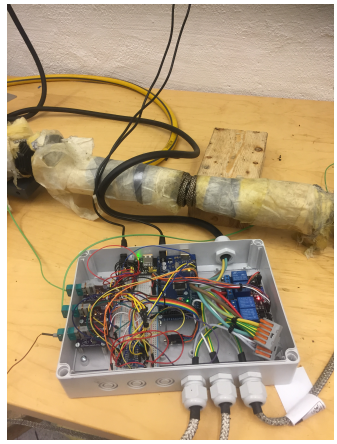


Figure 3.4: PID, for the control of heating tapes

3.2 Fluids

Marcol52 is a purified mixture of liquid saturated hydrocarbons and is used to simulate the oil phase. The density of the Marcol52 is $840kg/m^3$, and the viscosity is $6.9mm^2/s$ at 40 degrees Celsius. Due to several refining stages the lighter hydrocarbon components are removed, and thus no components will vaporize in ambient conditions, leaving the chemical composition constant. To be sure that no components will evaporate, a certain amount of pure Marcol52 was weighed, and heated over a 24hour span, and afterwards weighed again. The result showed that no components had evaporated.

To reproduce wax molecules, Sasolwax5603 is added to the oil phase in the heating tank, resulting in a oil-wax mixture with 10wt% Sasolwax5603 dissolved. The density of the Sasolwax5603 is $950kg/m^3$, and the composition is shown in figure 3.5

In case of large wax depositions, the amount of wax in the heating tank will be less than 10wt%. However, the tank contains 44 kg of oil with 4.4 kg of wax dissolved, and it is not expected that the deposition during a test will be more than 0.3kg. In this case, the amount of wax will change to 9%, and it is assumed that this change in composition has negligible effects on deposition pattern.

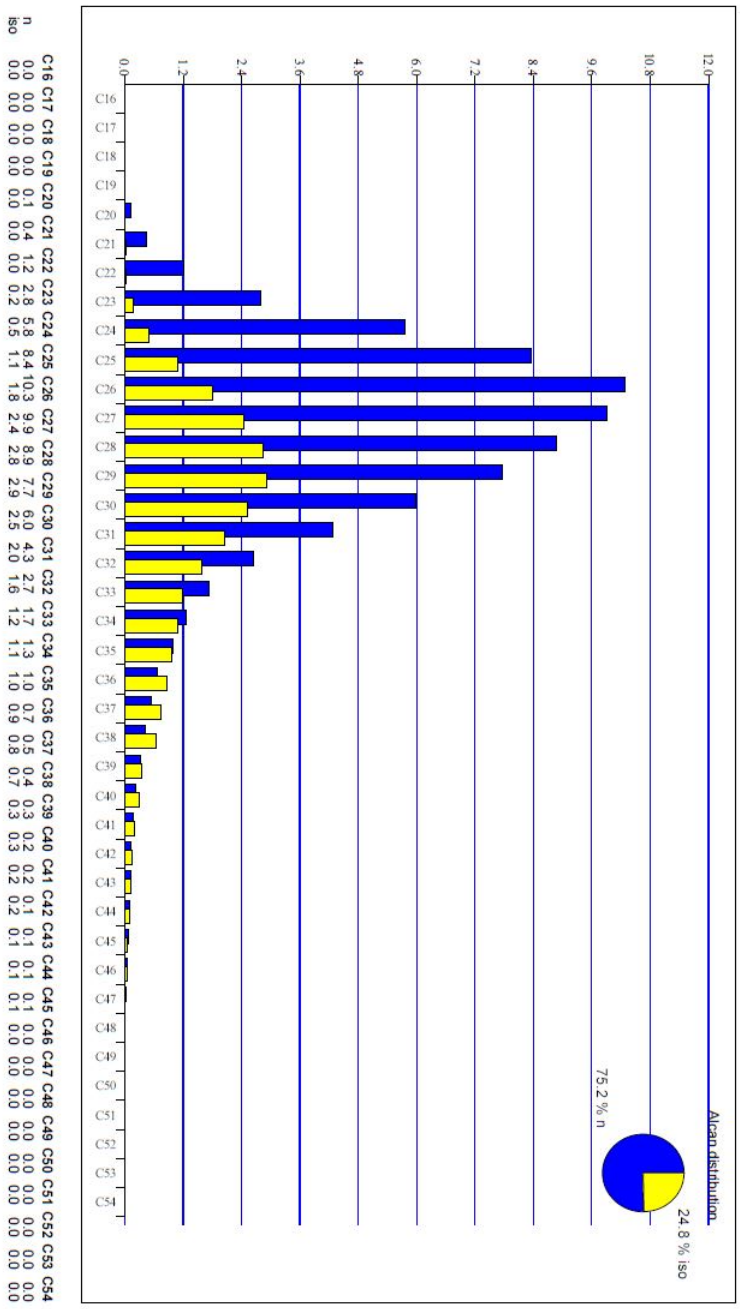


Figure 3.5: Wax Composition Sasolwax5603

3.3 Determination of WAT

As discussed in the theory part, several methods have been proposed to determine the WAT. At EPT a viscometer, sometimes called viscosimeter, is available for conducting experiments, and is shown in figure 3.6

Figure 3.6: Viscometer used to determine WAT of the waxy oil

The viscometer at NTNU was used to determine the WAT of the oil mixture. The test was divided into two parts. Firstly, a small sample of pure Marcol52 was placed on the viscometer, and the rotating plate was lowered to 300 micrometres above the base, forcing the rotating plate to be totally in contact with the oil. The viscometer has the possibility to change the temperature of the fluid and the shear rate applied to the fluid. The pure oil sample was heated to 80 degrees Celsius and cooled 0.5 degrees per minute until 5 degrees with a constant shear rate of 800 rounds per minute, and the viscosity was continuously logged. After the sample was cooled to 5 degrees, it was heated back to 80 degrees again, and the experiment was repeated to avoid errors.

Part two of the test was to repeat the experiment with Marcol52 with 10wt% Sasolwax5603 dissolved, and afterwards compare the two results. Where the viscosity vs temperature curve of the waxy oil differed from the pure oil curve, was determined as the WAT. Most likely the true WAT is higher than the measured WAT because it is required a significant amount of wax crystals to make an impact on the viscometer. However, this is considered as an uncertainty, and will not be considered further.

3.4 Drawing of Flow Map

The test rig at NTNU has two inner pipe possible setups for the test section, one with a copper pipe and one with a transparent acrylic pipe. The cobber pipe has the advantage of large thermal conductivity leading to rapid deposition of wax due to the cooling from the water. However, the cobber pipe is not transparent, and knowledge about flow regime must be based on indirectly measurements. The acrylic pipe has a very low thermal conductivity, leading to minimal cooling of the flowing waxy oil, thus minimal wax deposition. But the transparency of the pipe allows visual observations of the flow regime.

For this experiment, the visual observations method was chosen to determine the dominant flow regime. The waxy oil and gas was run through the transparent acrylic pipe with different u_{sl} and u_{sg} without cooling from the water, and the dominant flow regime of each run was determined by visual observations. For sufficient low u_{sg} and increasing u_{sl} the transition zone between stratified and slug flow is short and well defined. For larger u_{sg}, there exists a larger region of wavy stratified flow before the flows enter the slug flow region. As this thesis focus on the transition zone from stratified to slug flow, a u_{sg} yielding a sharp transition was chosen.

As the test was run without cooling, there was obviously not seen any deposition, and this kept the cross-sectional area of the test section constant. During an experiment with cobber pipe and cooling, the wax deposition will lead to a decrease in effective pipe diameter, which eventually could alter the flow regime. To cope with this challenge, the flow regime in the cobber pipe was visually confirmed as the expected regime using the $0.5meter$ transparent pipe section between mixing section and test section. In case of deviation from the expected flow regime, the flow map would be updated.

3.5 Procedure of the experiment

Before starting the experiment, the oil was heated to 60 degrees Celsius in the heating tank to dissolve the wax. The copper pipe and the flexible hoses normally have some wax from previous experiments, so the heated waxy oil and air was run through the test section for 30 minutes to ensure all wax was melted and dissolved in the oil. Thermocouples situated on the copper pipe were used to make sure the temperature in the copper pipe was well above the WAT.

To measure the wax deposition thickness, the pressure drop over the clean copper pipe had to be known. Depositions in the copper pipe was cleaned by the flowing fluids, and then the oil was flushed out with air. The air flow was then adjusted to the air flow always used during the experiments, and the pressure drop across the test section was noted.

After the pressure drop was noted, the oil flow was started again at the desired rate. Thereafter, cold water was added into the annular space between the copper pipe and the larger acrylic pipe. Water was added until it filled the entire annular space, and then the loop was closed to allow for the use of water pump. The water pump also acted like a

cooler, where it pumped 17 l/min of 5 degrees Celsius water around the annular space. When the water pump started, the experiment was run for two hours. The oil was held constantly at 60 degrees Celsius by heating in the tank and from the heating tape at inlet side of test section. During the experiment, the pressure drop across the test section, rate of the fluids, and temperatures along the test section were continuously logged.

After two hours the oil flow was stopped, and the air flow was increased to flush out all remaining oil in the pipe section. It was assumed that after two hours of testing, the deposition would be hard and not be affected by the increased air rates during flushing. After the oil was flushed out, the air flow was decreased to the rate previously used, and the new pressure drop across the pipe section was noted.

Although attempts were made to determine wax thickness indirectly through pressure drop measurements, the most accurate observations are achieved directly. After the pressure drop was measured, the water in the annulus was removed, and the copper pipe was dismantled from the stand. The pipe was weighed at an exact weigh, and as the weight of the clean pipe was known, the total weight of wax deposition could easily be calculated.

After measuring the wax deposition weight and volume, pictures were taken 15 cm inside the copper pipe with a boroscope. The boroscope was a 1200P 8LED IP68 Boroscope, where the LED lights made it possible to take picture of the deposition patterns inside the pipe.

Finally, the deposition volume was measured by addition of water into the pipe, as proposed by Matzain (1999). The cobber pipe was raised to vertical position, and a lock was placed on the end of the pipe to avoid water leaking out. Water was then added into the pipe, and since the volume of clean pipe without deposition was known, the volume of wax was easily calculated. Although the measurements of wax deposition using water yields repeatable results, the measurements are prone to human errors, and a volume measurement error of ± 5 millilitres was considered for the error margin.

3.5.1 Repeated procedure

A repeated list of the different steps already discussed above are listed below, and should always be followed:

1. Heat tank to 60 degrees Celsius, and then run the waxy oil for 30 minutes. Confirm visually that no wax is present in oil. Confirm that cobber pipe temperature is above WAT, to make sure all wax is melted
2. Stop oil flow, flush with air, adjust the air flow to desired rate, note the pressure drop across the test section after 60 seconds
3. Start oil flow
4. Add water in the annulus of the test section, and start the water pump
5. Run the experiment for two hours
6. After two hours, stop the oil flow, flush with air for 30 seconds, adjust the air flow to the same rate as during experiments, note the pressure drop
7. Drain water, dismantle the connections to remove the cobber pipe
8. Weigh the pipe to find deposition mass, take pictures with boroscope 15cm inside the pipe on both inlet and outlet side, and add water to find the volume of the wax deposition

Chapter 4

Results

4.1 Determination of WAT

The viscosity vs temperature of pure Marcol52 was measured, and the graph showed an almost linear increase of viscosity with decreasing temperature. The result is shown in figure 4.1 .

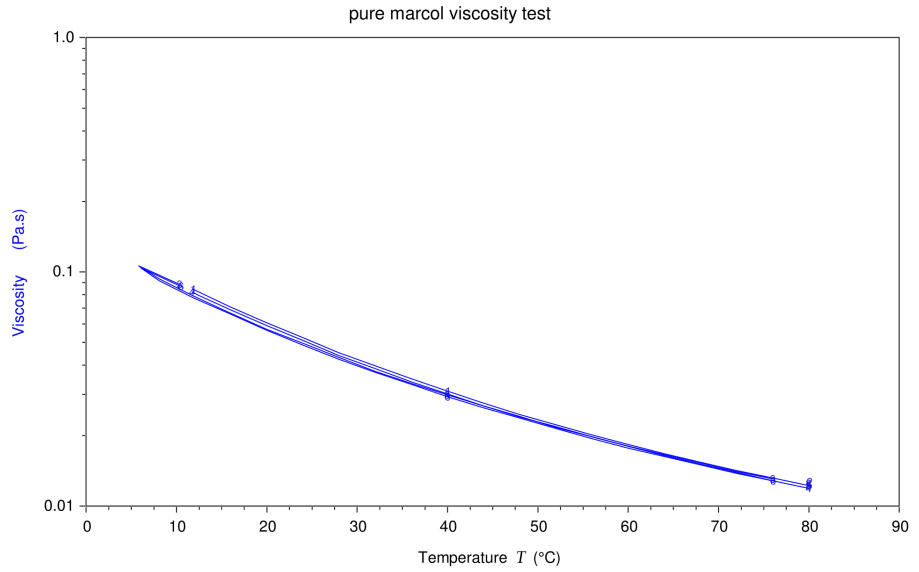


Figure 4.1: Viscosity vs temperature for pure Marcol52

Afterwards, a similar test was run with the mixture of Marcol52 and Sasolwax5603. The test conditions were similar, and the resulting graph seen in figure 4.2 shows that at approximately 33 degrees Celsius the viscosity starts to increase rapidly,

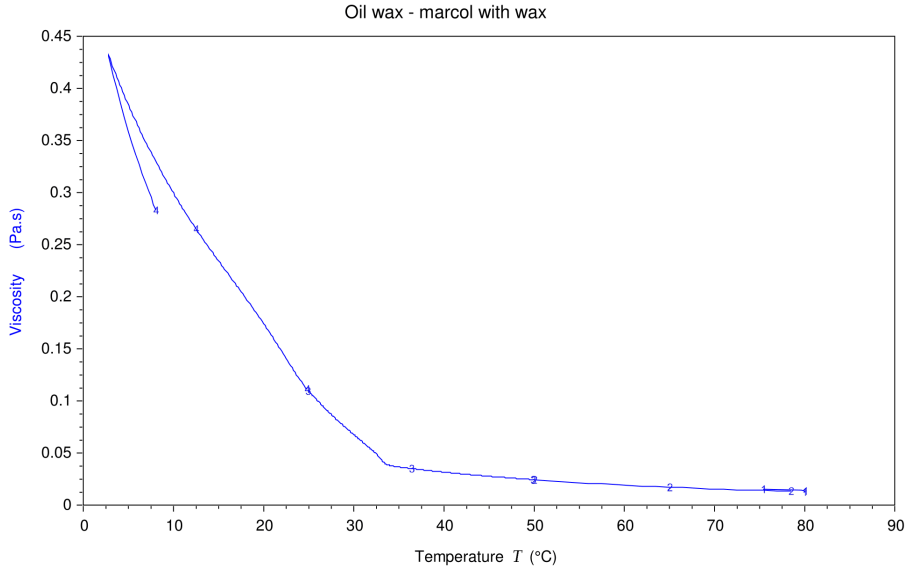


Figure 4.2: Viscosity vs viscosity for oil and wax mixture

4.2 Flow Map

After running the wax loop with several u_{sg} and u_{sl}, the dominant flow regime was determined by visual observations through the transparent acrylic pipe, and the resulting flow map is shown in figure 4.3

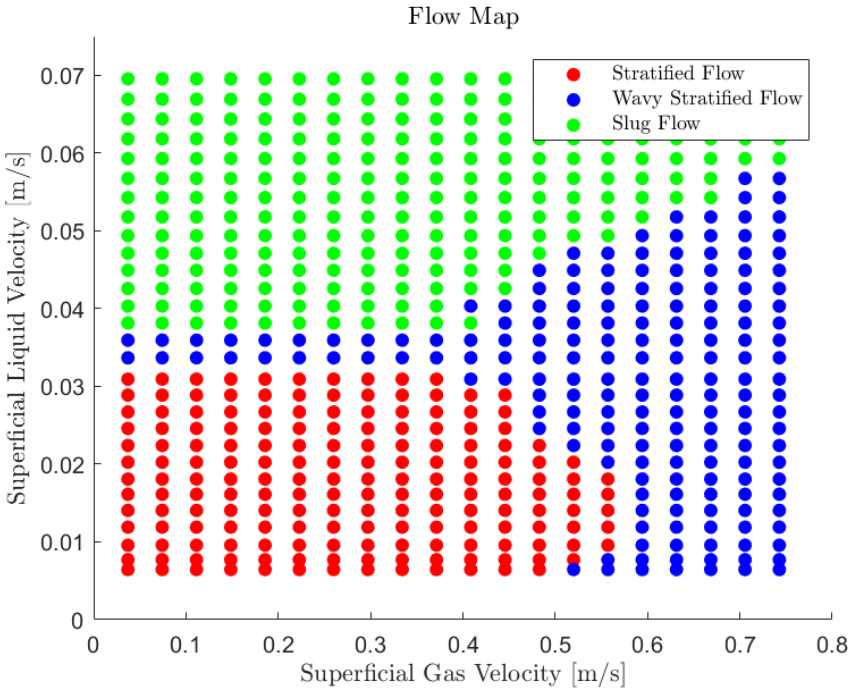


Figure 4.3: Flow Map

As seen from the flow map, the transition from stratified to slug flow is sharpest with low superficial gas velocities. Based on this, $u_{sg} = 0.22$ was chosen as constant value for all future experiments, and only the u_{sl} will be varying.

4.3 Wax deposition

A total of 17 tests were run at the wax loop, five in the stratified region, five in the wavy stratified region, and seven in the slug region. For each run, the deposition thickness and wax porosity was measured, and picture were taken inside the copper pipe. Graphs and results for one of each regime follows, and the remaining results can be found in Appendix A.

4.3.1 Stratified region

For $u_{sl} = 0.021$ and $u_{sg} = 0.22$ yielding stable stratified flow, the pressure drop across the test section was measured during the 2-hour test, and the resulting graph is shown in figure 4.8.

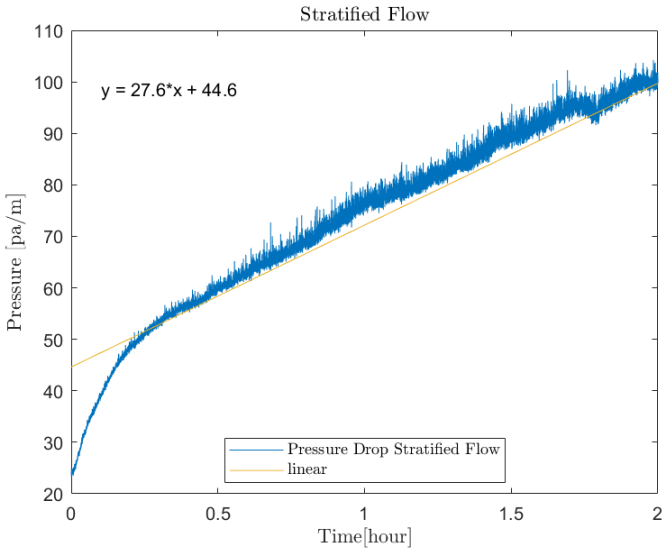


Figure 4.4: Pressure drop build-up for stratified flow

The linear trend line of $27.6x + 44.6$ shows that the pressure drop increases almost linearly during stratified flow.

4.3.2 The Effect of Cooling

The test was run for 10 minutes before cooling was started to look at the effects the addition of water has on the pressure drop. The temperature in the copper pipe was well above WAT without cooling but dropped rapidly below WAT when cooling was initiated. Figure 4.5 shows the increased pressure drop over the test section as the average temperature in the copper pipe dropped below WAT

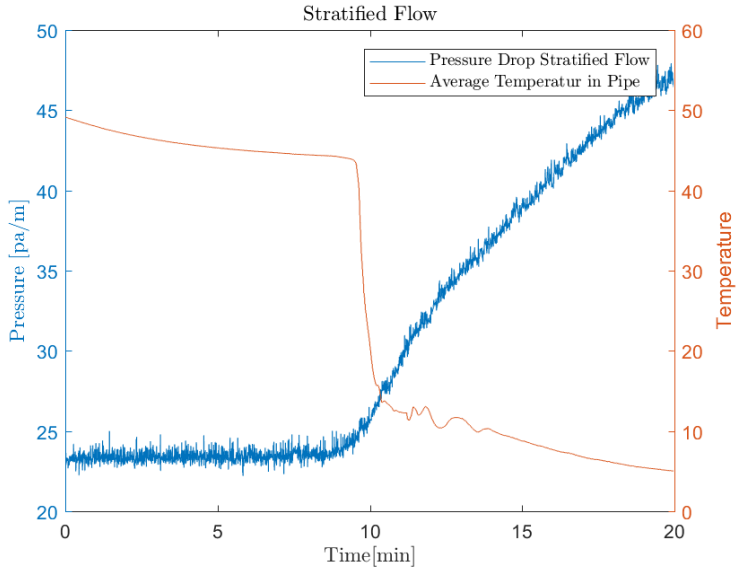


Figure 4.5: Pressure drop starts to increase due to wax deposition, because the average pressure drops below WAT

4.3.3 Pictures taken with Boroscope

Pictures were taken with a boroscope 15 cm inside the pipe on both inlet and outlet side. Figure 4.6 shows picture of the deposition pattern on inlet side

Figure 4.6: Picture 15cm inside copper pipe, inlet side

The deposition can be seen along sides of the pipe, and also as a flat river on the bottom of the pipe. The top side of the copper pipe has no deposition, which is proven by the red colour.

Figure 4.7 shows picture 15 cm inside the copper pipe on outlet side

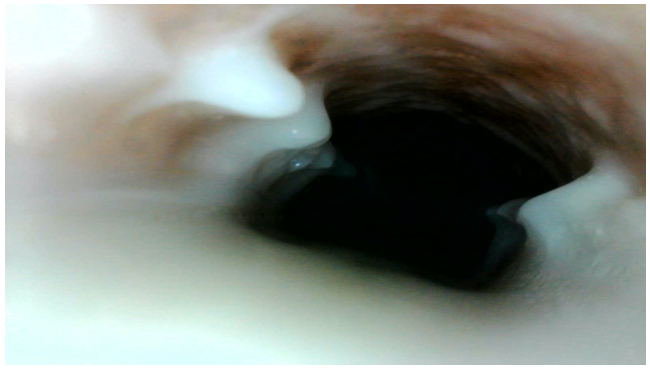


Figure 4.7: Picture 15cm inside copper pipe, outlet side

The deposition on outlet side is more disorganized than on inlet side. The deposition forms a river on the bottom of the pipe, but deposition along the sides are higher up on the pipe.

4.4 Slug Region

A total of 7 tests were run in the slug flow area. All the results are shown in Appendix A. For $u_{sl} = 0.04$ and $u_{sg} = 0.22$ yielding stable slug flow, 30 seconds of the experiment is shown in figure 4.8, and shows stable slug flow with slug frequency of 0.25/sec

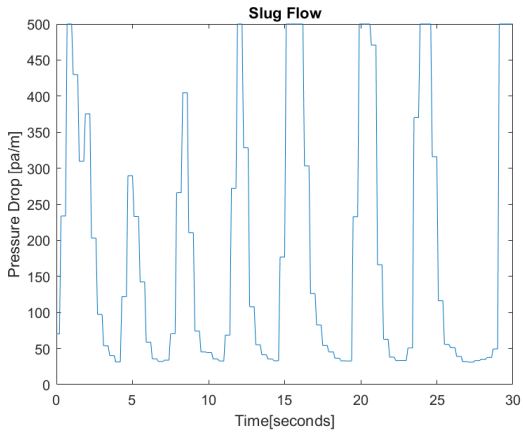


Figure 4.8: Pressure Drop over test section under Slug Flow

The trend line of the pressure build-up was determined to be $12x + 79$, indicating an increased pressure drop over the two-hour long test. However, the pressure build-up was lower than during stratified flow.

4.4.1 Pictures taken with Boroscope

Pictures where taken 15cm inside copper pipe with a boroscope on bot inlet and outlet side, and figure 4.9 shows the deposition on inlet side

Figure 4.9: Picture 15cm inside copper pipe, inlet side during slug flow

Figure 4.9 shows that the slug flows forms deposition along the circumference of the pipe. A small river of deposition is seen on the bottom part of the pipe, but for the rest of

the pipe the deposition is circumferential distributed inside the pipe.

Figure 4.10 shows the deposition at outlet side

Figure 4.10: Picture 15 cm inside copper pipe, outlet side during slug flow

The deposition pattern is similar on outlet side as inlet side, with deposition along the circumference of the pipe and a flat river of deposition along the bottom side of the pipe.

4.5 Wavy Stratified

A total of 5 tests were run in the wavy stratified region, and all the results are given in appendix A. For $u_{sl} = 0.027$ the increased pressure drop over time is given in figure 4.11

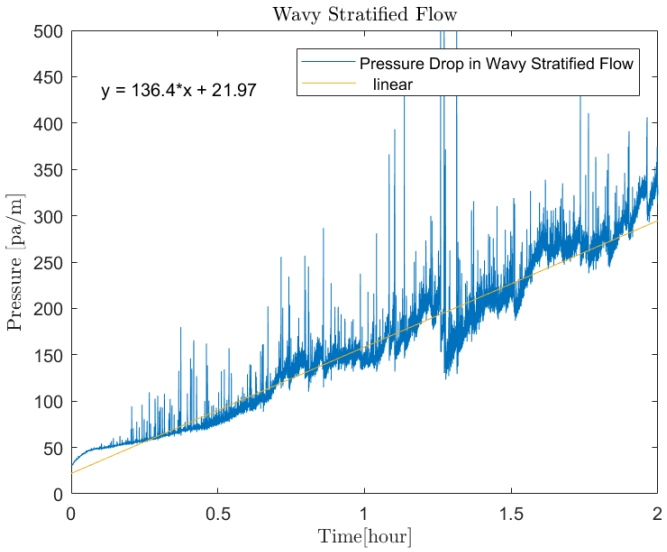


Figure 4.11: Trend line of increased pressure drop under Wavy Stratified Flow

The linear trend line of $136.4x + 21.97$ shows a constantly increase in pressure drop over time, significantly larger than seen under stratified and slug flow.

4.5.1 Pictures taken with Boroscope

Pictures were taken 15cm inside the copper pipe on inlet and outlet side to determine deposition patterns. 4.12 shows deposition on inlet side

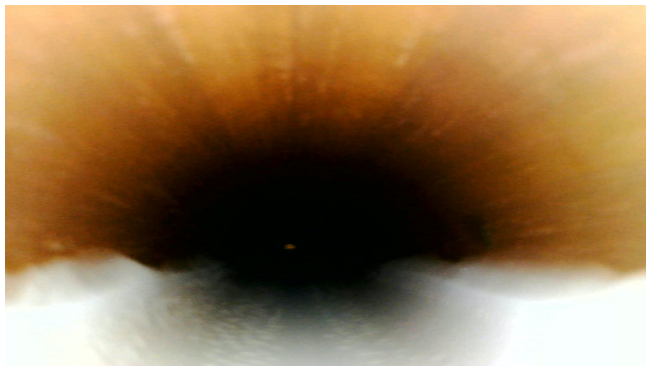


Figure 4.12: Picture taken 15cm inside copper pipe, inlet side under wavy stratified flow

Figure 4.12 shows similar deposition pattern at inlet side as under stratified, with a flat deposition river on bottom of pipe, and no deposition on the top side of the pipe.

Figure 4.13 shows deposition at outlet side

Figure 4.13: Picture taken 15cm inside copper pipe, outlet side under wavy stratified flow

Figure 4.13 shows the deposition forming a flat river on the bottom side of the pipe and a thicker deposition on the top side of the pipe.

4.6 Wax Thickness

The wax deposition thickness was determined using the Spool Piece Method, where the test section was dismantled after each run to measure the weight and volume of the deposition. A total of 17 tests were run, 5 in the stratified region, 5 in the wavy stratified region, and 7 in the slug flow region. The resulting table is shown below in table 4.1, and the plotted wax thickness vs superficial liquid velocity is shown in figure 4.14

Run	u_{sl}[m/s]	u_{sg} [m/s]	m_{dep} [kg]	ρ_{dep}	Flow Regime	Wax Thickness [mm]
1	0.007	0.22	0.2258	898.5	Stratified	1.71
2	0.074	0.22	0.1946	920.1	Slug	1.42
3	0.035	0.22	0.2896	899.5	Slug	2.25
4	0.039	0.22	0.2276	889.8	Slug	1.75
5	0.027	0.22	0.3663	842.8	Wavy Stratified	3.16
6	0.053	0.22	0.2419	888.4	Slug	1.87
7	0.024	0.22	0.3735	871.9	Wavy Stratified	3.11
8	0.033	0.22	0.2537	882.0	Slug	1.99
9	0.013	0.22	0.3085	884.2	Stratified	2.46
10	0.018	0.22	0.3173	889.9	Stratified	2.52
11	0.021	0.22	0.3187	895.6	Stratified	2.51
12	0.028	0.22	0.3536	859.7	Wavy Stratified	2.97
13	0.009	0.22	0.2653	903.9	Stratified	2.03
14	0.029	0.22	0.3577	889.6	Wavy Stratified	2.89
15	0.031	0.22	0.3049	897.4	Slug	2.39
16	0.041	0.22	0.2096	888.4	Slug	1.60
17	0.026	0.22	0.3863	949.5	Wavy Stratified	3.34

Table 4.1: Wax Thickness Results

Figure 4.14 shows an increase in deposition thickness for increased u_{sl} during stratified flow. The deposition thickness under wavy stratified flow is higher than in stratified and slug flow, and almost constant. For slug flow the deposition is decreasing with increasing flow rate.

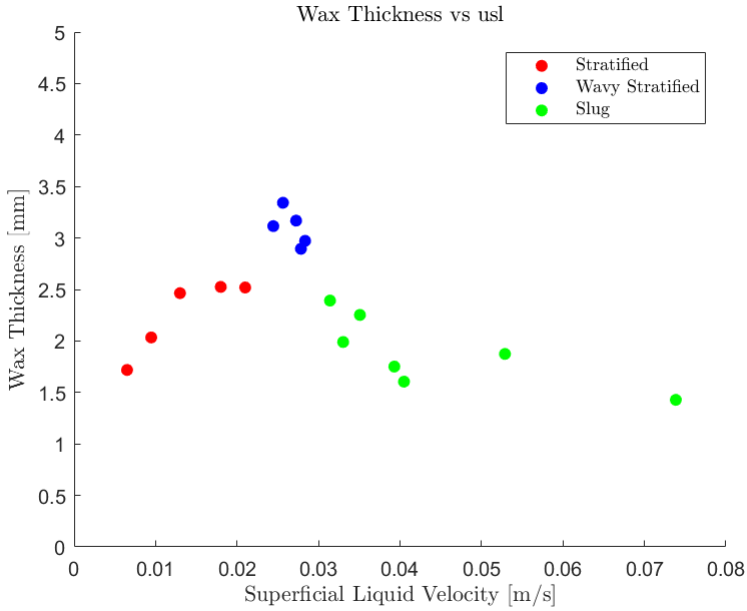


Figure 4.14: Wax Thickness deposition thickness for stratified, wavy stratified and slug flow

Figure 4.15 and 4.16 show the same graph with trend line for stratified and slug flow

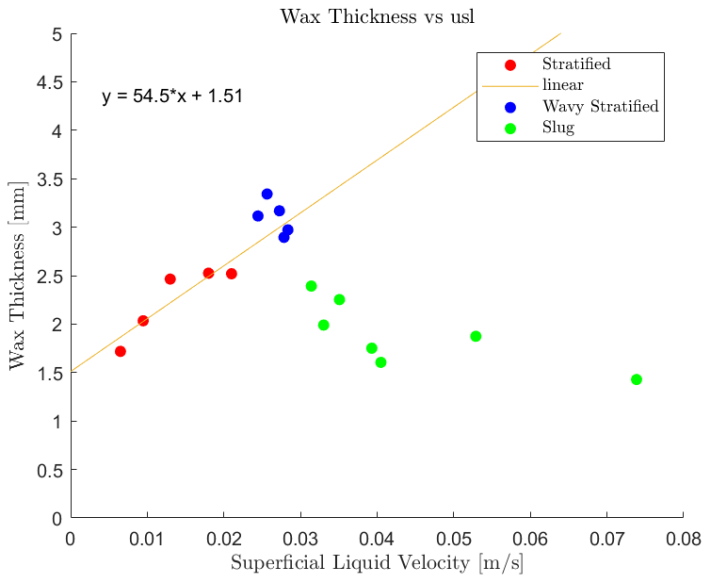


Figure 4.15: Linear trend line for stratified flow

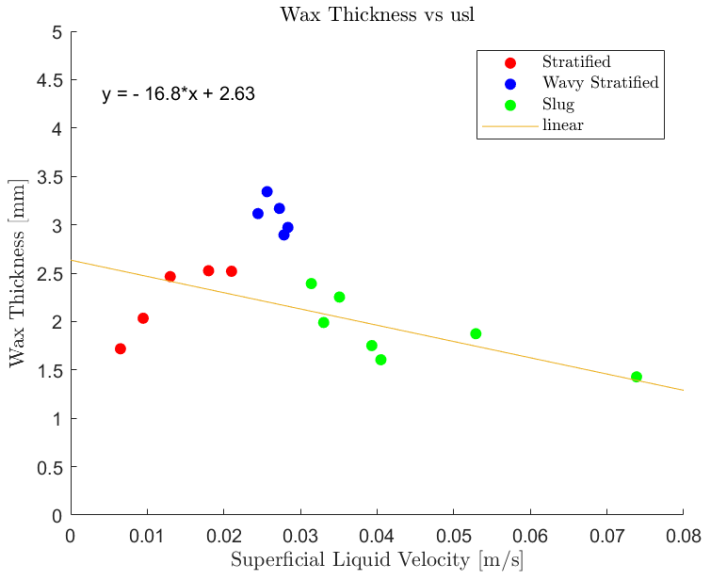


Figure 4.16: Linear trend line for slug flow

4.7 Effects of Liquid Holdup

The Holdup was predicted using the program MultiPhasePlot, and the result is shown in figure 4.17

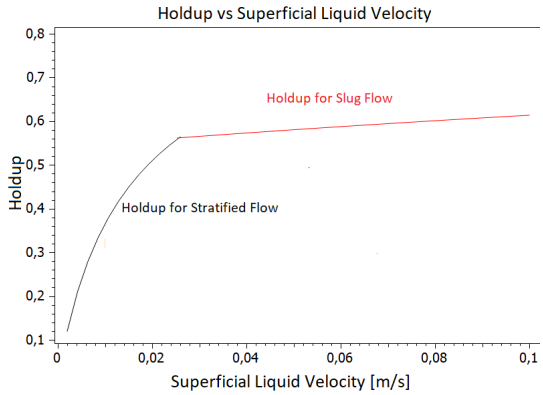


Figure 4.17: Calculated Holdup for stratified and slug flow

The flow is expected to be stratified until $u_{sl} = 0.025$ with an increase in holdup with increasing u_{sl}. The red line represents the holdup factor in stratified flow, and the black line in slug flow. Slug flow is expected after $u_{sl} = 0.025$, and the holdup factor increases linearly with increasing u_{sl}.

The values from MultiPhasePlot were imported to MatLab and plotted against wax thickness deposition in Matlab, and the result is shown in figure 4.18

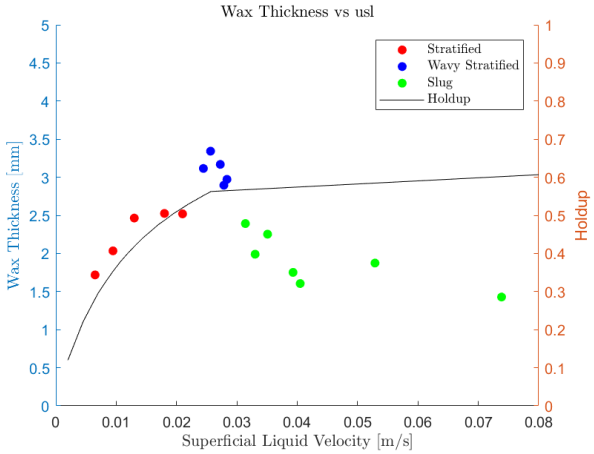


Figure 4.18: Wax Thickness and Holdup vs usl

4.8 Wax Thickness Determined from Pressure Drop

The wax thickness was determined using The Pressure Drop Method explained in Chapter 3, and the resulting wax thickness is shown in figure 4.19

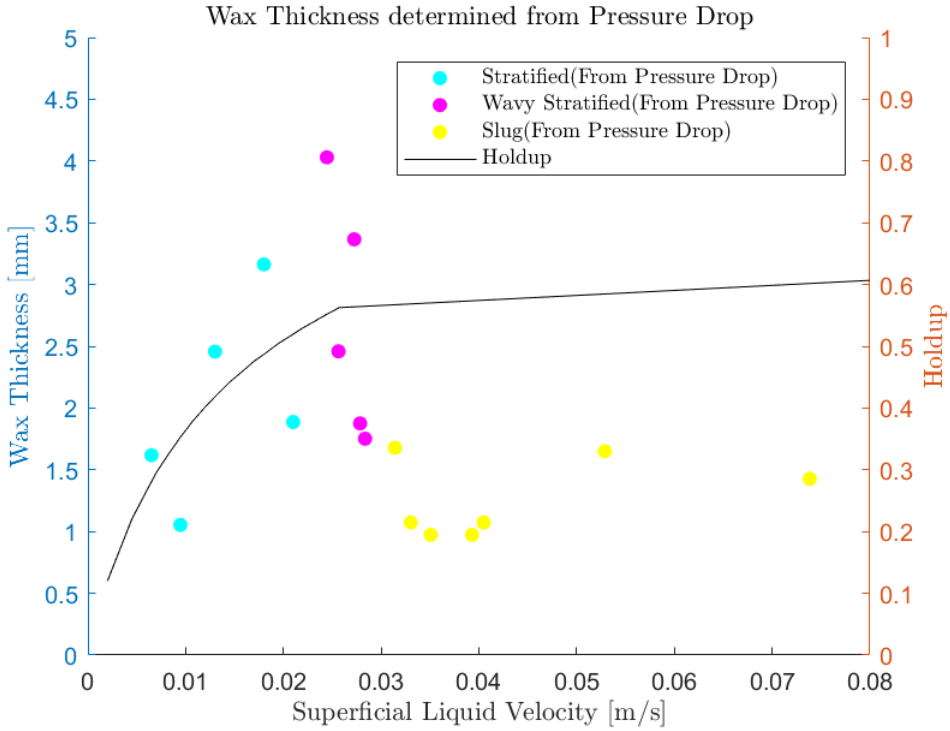


Figure 4.19: Holdup and Wax Thickness Determined from Pressure Drop

The wax thickness determined from The Pressure Drop Method was compared to the thickness determined using The Spool Piece Method, and the resulting graph is shown in figure 4.20

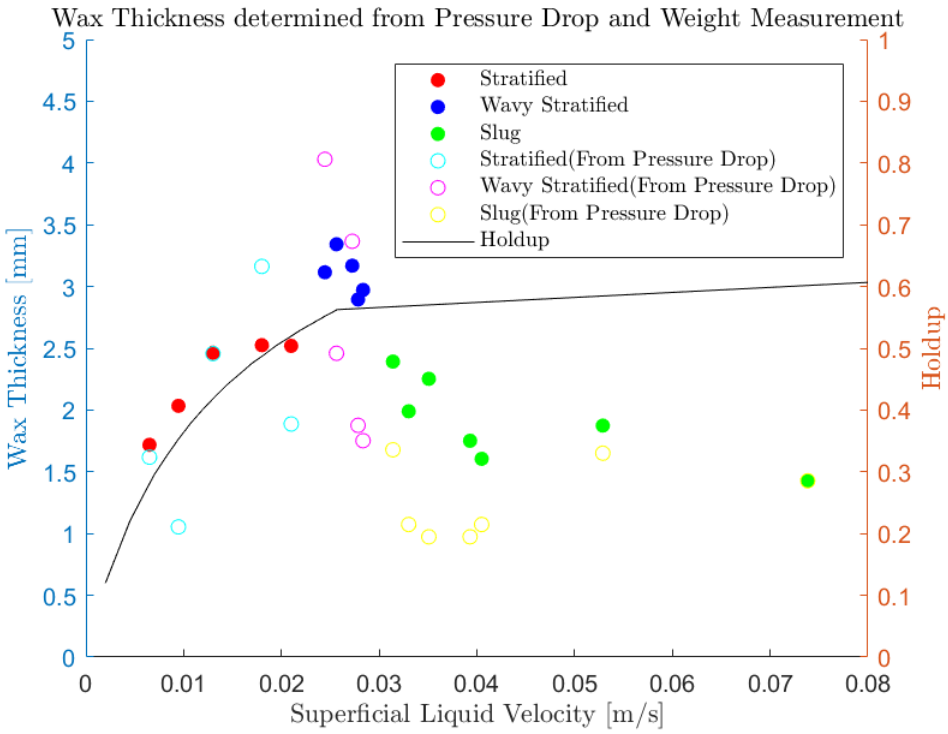


Figure 4.20: Comparison of wax thickness determination using Spool Piece and Pressure Drop Method

4.9 Wax porosity

The wax porosity was measured after each run by adding water to the pipe to find the wax volume. The wax porosity was then easily back calculated, and figure 4.21 shows the resulting graph

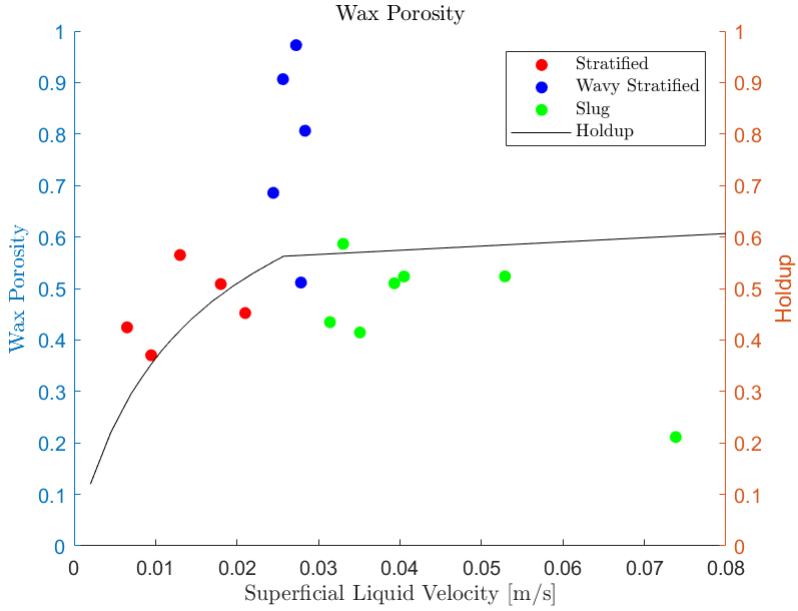


Figure 4.21: Wax porosity for different liquid superficial velocities

4.10 Uncertainty Calculations

When measuring the volume of the pipe with wax deposition, it is assumed an uncertainty of ± 5 ml of added water. This uncertainty gives the average percentage error of the wax porosity shown in the table 4.2

Run	Original ϕ	ϕ+5 [ml]	ϕ -5 [ml]	Average Error [%]
1	0.425	0.649	0.188	54.2
2	0.211	-0.037	0.446	114.5
3	0.415	0.569	0.259	37.3
4	0.510	0.667	0.348	31.3
5	0.973	1.087	0.856	11.9
6	0.524	0.703	0.338	34.8
7	0.686	0.795	0.570	16.4
8	0.587	0.756	0.413	29.2
9	0.565	0.714	0.411	26.8
10	0.509	0.622	0.394	22.4
11	0.452	0.595	0.305	32.1
12	0.806	0.929	0.680	15.4
13	0.370	0.539	0.195	46.5
14	0.511	0.663	0.355	30.1
15	0.435	0.580	0.285	33.9
16	0.523	0.729	0.308	40.2
17	0.907	1.011	0.801	11.6

Table 4.2: Percentage Error of ϕ

For the wax thickness measurement using The Spool Piece Method, the density of the deposited wax must be calculated using the wax porosity. The uncertainty in wax porosity due to the addition of water into the pipe after testing, yields the percentage error of wax thickness shown in table 4.3

Run	Original δ	δ +5 [ml]	δ -5 [ml]	Average Error [%]
1	1.718	1.767	1.670	2.8
2	1.428	1.469	1.388	2.8
3	2.253	2.297	2.209	1.9
4	1.751	1.786	1.717	1.9
5	3.169	3.221	3.117	1.6
6	1.874	1.917	1.832	2.3
7	3.116	3.164	3.067	1.6
8	1.990	2.033	1.947	2.2
9	2.464	2.513	2.416	2.0
10	2.525	2.562	2.488	1.5
11	2.519	2.566	2.473	1.8
12	2.972	3.023	2.922	1.7
13	2.034	2.077	1.991	2.1
14	2.895	2.954	2.837	2.0
15	2.392	2.437	2.347	1.9
16	1.605	1.647	1.564	2.6
17	3.342	3.392	3.293	1.5

Table 4.3: Percentage Error of δ

Chapter 5

Discussion

The objective of this master thesis was to investigate how different flow regimes and increased holdup affect the wax deposition thickness and wax porosity, and to investigate whether the Pressure Drop Method developed for single phase flow can be used in multiphase flow. In addition, which mechanisms that contributes to deposition during multiphase flow was also investigated.

5.1 Spool Piece Method vs Pressure Drop Method

Two methods were used to determine the wax thickness deposition after each run. The two methods were namely

- Spool Piece Method
- Pressure Drop Method

The Spool Piece Method has the advantage that is a direct method were the results are based on actual observations such as weight and volume measurements. However, the method has some disadvantages. The wax thickness determined from the Spool Piece Method is an average thickness across the pipe section, and the wax thickness at a specific location at the pipe is not possible to determine. Therefore, the actual wax thickness at the outlet of the pipe is expected to be higher than the thickness calculated. The magnitude of this deviation depends on the wax distribution along the test section, whether it is uniformly or not. However, it is assumed that the wax deposition is relatively uniformly distributed along the test section, so that the thickness calculated from The Spool Piece Method is equal to the real wax thickness.

Based on the results presented in figure 4.20, it is clear that the wax thickness determined using The Pressure Drop Method does not corresponds to the results determined using The Spool Piece Method. For most of the runs, the wax thickness determined from The Pressure Drop Method was significantly lower than from The Spool Piece Method.

However, this is not consistence, as some of the values are higher than the expected ones. The main uncertainty is the assumption that the friction factor is equal with flow in clean copper pipe and in pipe with wax deposition, and most likely this is the source of the errors. If the friction factor with a wax layer with unknown thickness could be exactly estimated, then the Pressure Drop Method could probably be used. Further studies should be conducted.

Based on these discussion, only the wax thickness determined from The Spool Piece Method will be considered further.

5.2 Wax Thickness

Figure 4.18 shows that the wax deposition thickness during stratified flow is increasing with increasing holdup. This is very logical, all the time the wax only deposits on the wetted part of the pipe. However, the deposition thickness is slightly larger than the holdup curve, for most of the values. This can be explained by the possibility that the interface between air and oil is not flat. The interface can be concave, thus allowing more of the pipe to be wetted than the holdup factor implies. This was not studied in this thesis but should be further investigated.

Another explanation could be that when the first wax starts to deposit on the bottom of the pipe, the effective diameter of the pipe decreases. Thus, the holdup factor calculated using a clean pipe will be too low, and the wax deposition thickness calculated reflects this new effective holdup. This first deposit acts like an insulator preventing further deposition in the bottom part of the pipe and allows the flowing waxy oil to wet a larger section of the circumference of the pipe. If this is true, the same effect should be seen during wavy stratified flow.
For some of the pictures taking inside the copper pipe, there can be seen some wax deposition on the top side of the copper pipe on outlet side. This occurred most likely not during the test, but rather during flushing of the pipe after experiments was finished, where the increased gas rate and decreased holdup as the oil was removed made some of the oil particles to hit the wall as droplets.

When the multiphase flow entered the wavy stratified region, the wax deposition thickness increased significantly. Figure 4.18 shows this clearly, where the average wax thickness of the five wavy stratified runs are 3mm. During stratified flow the slope of the thickness increase was almost linear, following the holdup factor curve, but when entering the wavy stratified region, the deposition thickness increased more than the increase during stratified flow. This was because wax deposited both on the bottom and top parts of the pipe. The deposition on the bottom part of the pipe is similar as in stratified flow, with a deposition river on the bottom. The deposition on the top part of the pipe is due to waxy oil particles leaving the stratified flow as droplets and hitting the cold top of the copper pipe where it instantaneously precipitates and deposits as a thick waxy gel. Because the top part of the pipe was not constantly wetted by the flowing fluid, the deposit experienced minimal aging and shear removal effect compared to the deposition on the bottom. Thus,

the deposition on the top part of the pipe is softer and thicker than on the bottom part, and this was also confirmed by pictures taken inside the pipe.

After the flow entered the slug region, the deposition thickness drastically decreased. Even though the holdup curve continued to increase in the slug region, the deposition thickness decreased towards 1.5mm for $u_{sl} = 0.053$ compared to 3mm in the wavy stratified region. This was due to the shear removal effects removing some of the wax already deposited. The shear stress exerted by the fluid flow at the deposit interface was large enough to mechanically remove some of the deposit already formed on the pipe wall. Even though the holdup increased, the deposition thickness decreased. The thickness did not decrease to zero. Due to the high thermal conductivity in the copper pipe, the wall was always close to the temperature of the surrounding water, namely 5 degrees Celsius. At this temperature wax deposited on the wall, and if the shear forces were large enough to remove this deposit, new deposits were formed.

5.3 Wax Porosity

Another objective of this thesis was to investigate the effect flow regimes and increased holdup had on the wax porosity of the deposited layer. The results presented in figure 4.21 are not as clear as for the wax thickness. The graph shows higher wax porosity for depositions during wavy stratified flow than in stratified and slug flow. The wax porosity in the stratified region are somewhat consistent in the region between $0.35 - 0.6$. It was expected that the wax porosity in the stratified region would be constant, as the deposition pattern was the same with deposition only along the bottom part of the pipe, and no large shear forces were seen. After the wax was deposited, the low aging effect should not result in a hardening of the deposition, but the results show varying wax porosity. The results may be unreliable due to errors in measurements.

For the wavy stratified region, the wax porosity was the highest with values ranging from 0.5 to 0.9. From pictures taken inside the pipe of the deposition pattern, it is clear that the deposition occurred at both bottom and upper parts of the pipe. In case of a droplet hitting the cold top side of the pipe, and all the oil was trapped inside the wax crystal structure, the wax porosity would be 0.9, the same as the oil/wax ratio. The aging effect on the bottom part is expected to be close to similar as for the stratified region, but for the top part it is expected to be lower, since it was not continuously wetted. Thus, the total wax porosity will be an average of the wax porosity in the bottom and top part. However, the resulting wax porosity during wavy stratified flow has a very wide range of values, and the reliability of the results must be discussed.

For the slug region, the figure 4.21 shows that the wax porosity is lower than during wavy stratified, and somewhat similar as during stratified flow. The high shear stress exerted by the fluid was expected to encourage aging effects, where the oil trapped inside the deposition layer will diffuse out of the crystal structure, leaving the depositions with a low wax porosity. The resulting graph shows that the wax porosity is lower than during

wavy stratified flow, which could be due to the ageing. However, there is not seen a significantly decrease in wax porosity with increasing shear forces, which was expected to happen. Thus, the results are challenging to interpret.

5.4 Gravity Settling

As discussed in Chapter 2, most researchers consider diffusion as the main deposition mechanism, and that deposition due to gravity settling is negligible. However, the pictures taken inside the copper pipes after each test indicates that wax may have been deposited due to gravity settling. If diffusion was the only deposition mechanism, the deposition would have been as illustrated in figure 5.1, and would be equally thick along the wetted part of the pipe for stratified flow, and equally thick along the entire circumference of the pipe for slug flow.

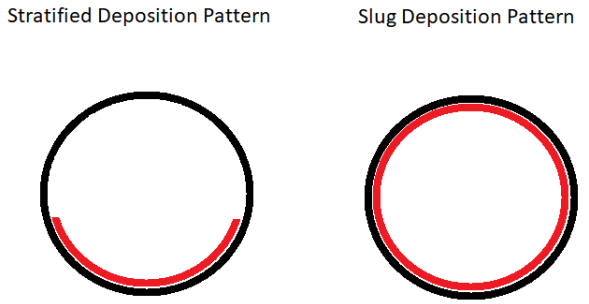


Figure 5.1: Expected deposition pattern for stratified and slug flow if diffusion was the only mechanism. The red part on the figure is the wax deposition.

However, the deposition pattern from the tests conducted during this theses clearly shows deposition patterns affected by the gravity, and is shown in figure 5.2

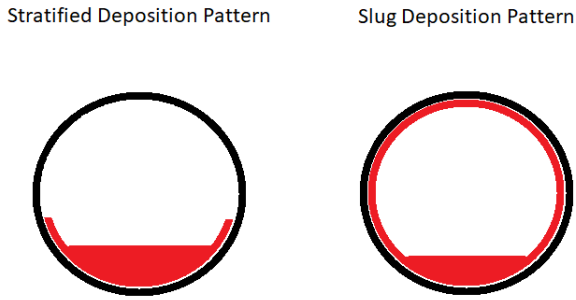


Figure 5.2: Deposition pattern found from this thesis for stratified and slug flow.

In stratified flow a flat deposition is seen along the bottom part of the pipe. Even in slug flow, where deposition is seen around the circumference of the pipe, a thicker deposition is seen on the bottom part of the pipe. These deposition patterns can only be explained by gravity settling, where the gravity force pulls on the heavier wax molecules in the flow. This flat deposition pattern is thicker in stratified flow than in slug flow, where the low superficial velocity gives the wax extra time to deposited due to gravity settling.

5.5 Uncertainties

The results in this thesis have some uncertainties. The largest uncertainty is that it is hard to exactly measure the volume of water added into the pipe when determining the wax porosity, and small deviations can lead to large errors. It was assumed an uncertainty of $\pm 5ml$ of added water due to human errors. Table 4.2 shows the average percentage errors in the range of 11.6% to 114.5%. Normally, a percentage error below 5% is assumed negligible. As all the tests have percentage errors above 5%, the results are unreliable, and should not be considered further.

Also, for the calculation of wax thickness using the Spool Piece Method, the volume of the pipe after each test had to be known. Again, it was hard to determine exactly the volume of added water into the pipe after each test. As table 4.3 shows, the assumed uncertainty of ± 5 ml of added water did not affect the calculated wax thickness significantly. The average percentage error ranged from 1.5% to 2.8%. As all the percentage errors are above 5%, the errors are negligible, and the results are assumed to be correct.

After each run, the pipe had to be flushed with air to remove all oil in the pipe. If the test section after each run was emptied of remaining oil identically, is an uncertainty factor. When the test ended, the oil flow was stopped, and the gas rate was raised to the air gas rate of 1.75 l/sec for 30 seconds to flush out remaining oil. If the pipe would not have been flushed, all the oil present at stopping time would deposit, thus resulting in too high wax deposition results. On the other hand, flushing with too high air rate can damage the

deposition. The specific air rate of 1.74 l/min was therefore chosen, where it was assumed to be large enough to empty the test section, but also low enough to not damage the wax already deposited.

It appeared that some gas was trapped inside the wax deposition and was gradually released when water was added. Figure 5.3 shows two bubbles of gas escaping out of the water when volume measurement was taken. It is not known how much impact this release of gas has on the wax porosity, and it is considered as an uncertainty.

Figure 5.3: Two Gas Bubbles rising out of the pipe section during volume measurements

Another uncertainty factor is the rig setup, where the repeatedly dismantling of the test section can alter the geometry and parameters of the flow system. At low u_{sl} and u_{sg} in horizontal pipes the dominant flow regime is very sensitive to deviation in angles. An upward incline of only 1% could change a flow from stratified to slug. Therefore, before every test a leveller was used and the height on each side of the test section was thoroughly examined to ensure horizontally oriented pipe section. However, small deviations can still occur, thus inducing uncertainties into the results.

Chapter 6

Conclusion

From the results presented in chapter four, and the discussion in chapter five, the main conclusions are the following

- Wax thickness is increasing with increasing u_{sl} during stratified flow, ranging from 1.5 mm to 2.5 mm, and the deposition is seen only on the bottom part of the pipe
- Wax deposition is thickest during wavy stratified, with an average thickness of 3 mm, due to wax deposits both on the bottom and top part of the pipe.
- Wax Thickness is decreasing with increasing u_{sl} during slug flow, ranging from 2.5 mm close to the wavy stratified region, until 1.5 mm. The shear stress exerted by the flowing fluid to the deposition is high enough to mechanically strip of already deposited wax.
- Wax Thickness measurement using The Pressure Drop Method is not applicable in multiphase flow.
- Deposition due to gravity settling occurred in stratified, wavy stratified and slug flow.

Chapter 7

Recommendation for Further Work

7.1 Experimentally

More experiments should be performed at the multiphase flow loop at NTNU to further investigate wax deposition thickness as a function of flow regime. As only 17 tests were conducted at this thesis, more experiments should be run to repeat the results, and to eliminate errors in the measurements. The method of added water proved to be very sensitive for human errors, and efforts should be put in to develop a more accurate measurement technique for the determination of wax porosity.

In addition, performing multiphase wax deposition experiments in a larger scale facility with larger diameter pipes and higher gas and liquid rates should be done. It should also be performed tests in high pressure system to reproduce reservoir conditions, and to investigated if these results would be similar as in the low-pressure system used in this thesis.

Deposition in multiphase flow due to gravity settling should be further investigated. The results in this thesis clearly shows deposition due to gravity settling. This conclusion contradicts the belief of most researchers, so further experimental studies needs to be performed to confirm these results. Experiments in the slug flow region conducted during this thesis should be repeated with the test section in vertical position, and deviations in the wax deposition patterns from vertical and horizontally oriented pipes indicate deposition due to gravity settling. If the liquid and gas rates used in this thesis are too low to achieve slug flow in vertical pipes, the rates should be increased and then performed in both vertical and horizontally oriented pipes.

7.2 Studies

Efforts should be put in to make the Pressure Drop Method applicable in multiphase flow. Indirect methods are more convenient to use than direct methods, where the test section

must be dismantled after every run. Experimental and literature studies should therefore be performed to develop a model for the prediction of the friction factor in multiphase flow with wax deposition.

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Appendix A

Additional Results

Run 1, Stratified Flow $usl = 0.007$

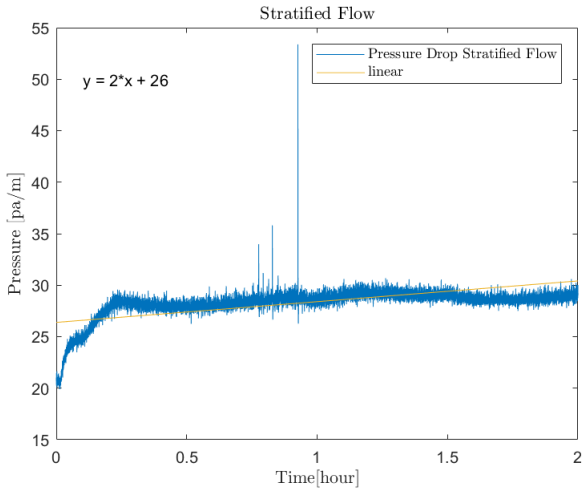

Figure 7.1: Pressure Drop increase over test section under stratified flow

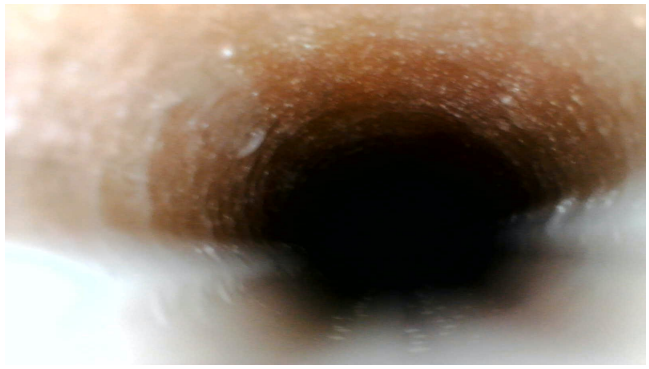

Figure 7.2: Picture taken 15cm inside copper pipe, inlet side

Figure 7.3: Picture taken 15cm inside copper pipe, outlet side

Run 2, Slug Flow $u_{sl} = 0.074$

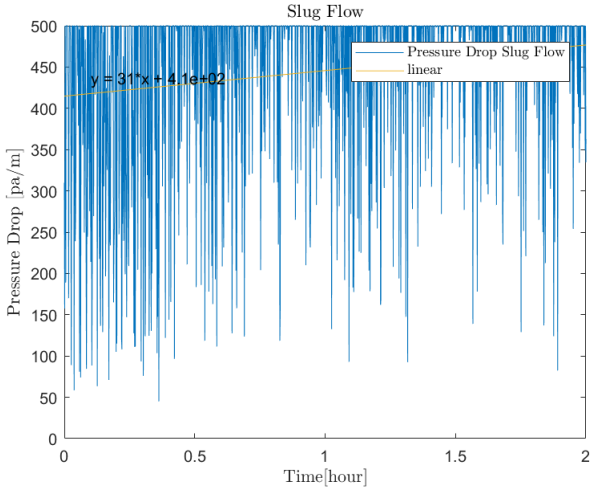

Figure 7.4: Pressure drop incerase over test section under slug flow

Figure 7.5: Picture taken 15cm inside copper pipe, inlet side

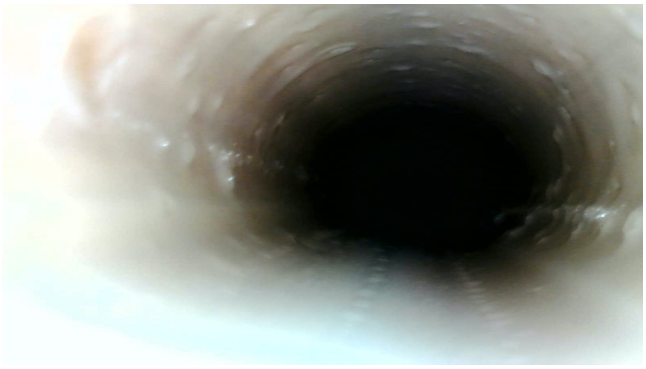

Figure 7.6: Picture taken 15cm inside copper pipe, outlet side

Run 3, Slug Flow $u_{sl} = 0.035$

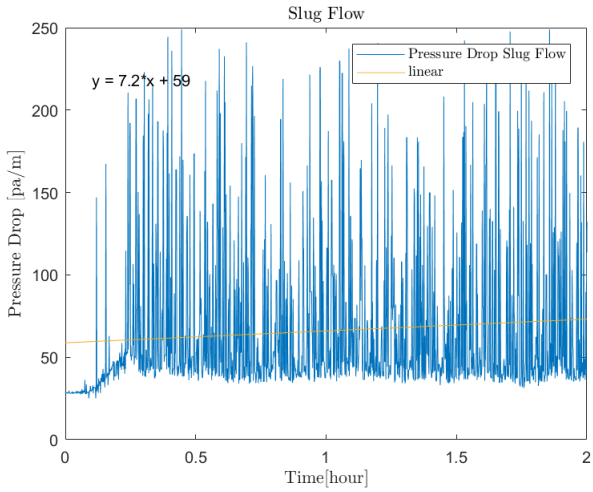

Figure 7.7: Pressure Drop increase over test section under slug flow

Figure 7.8: Picture taken 15cm inside copper pipe, inlet side

Figure 7.9: Picture taken 15cm inside copper pipe, outlet side

Run 4, Slug Flow $u_{sl} = 0.039$

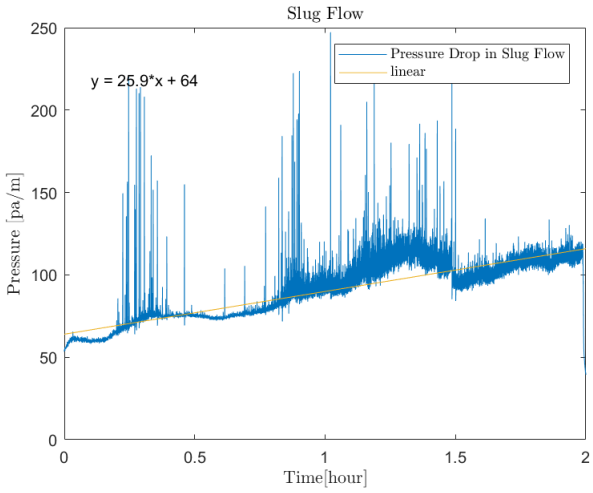

Figure 7.10: Pressure Drop increase over test section under slug flow

Figure 7.11: Picture taken 15cm inside copper pipe, inlet side

Figure 7.12: Picture taken 15cm inside copper pipe, outlet side

Run 5, Wavy Stratified $u_{sl} = 0.027$

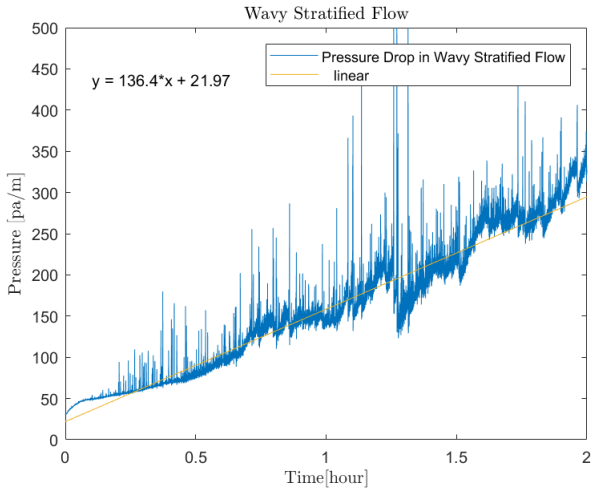

Figure 7.13: Pressure Drop increase over test section under wavy stratified flow

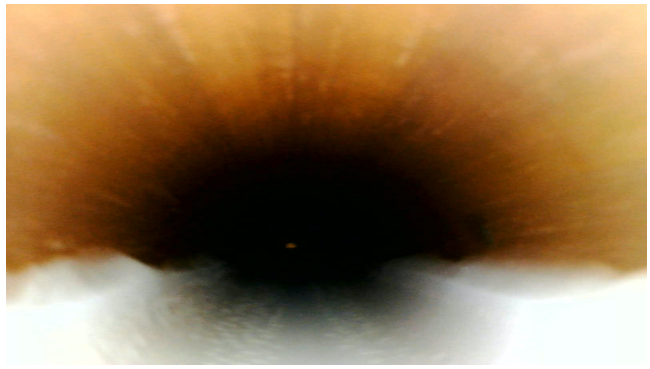

Figure 7.14: Picture taken 15cm inside copper pipe, inlet side

Figure 7.15: Picture taken 15cm inside copper pipe, outlet side

Run 6, Slug Flow $u_{sl} = 0.053$

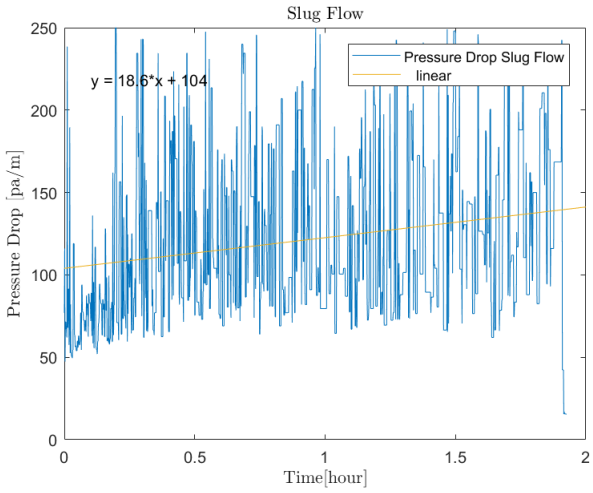

Figure 7.16: Pressure Drop increase over test section under slug flow

Figure 7.17: Picture taken 15cm inside copper pipe, inlet side

Figure 7.18: Picture taken 15cm inside copper pipe, outlet side

Run 7, Wavy Stratified $usl = 0.024$

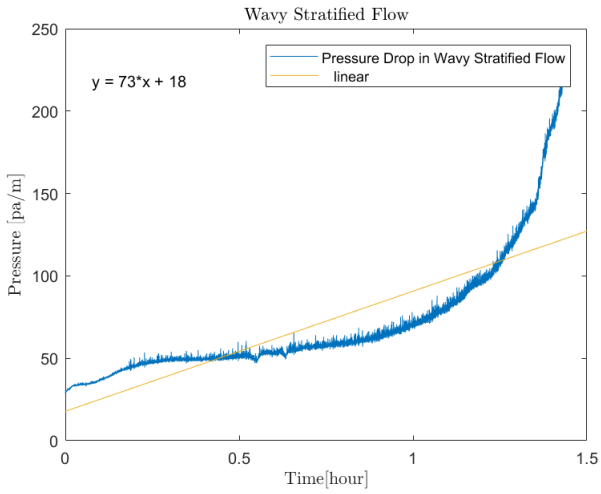

Figure 7.19: Pressure Drop increase over test section under Wavy Stratified flow. Test ended after 1.4 hours, but assumed to be valid for two hours

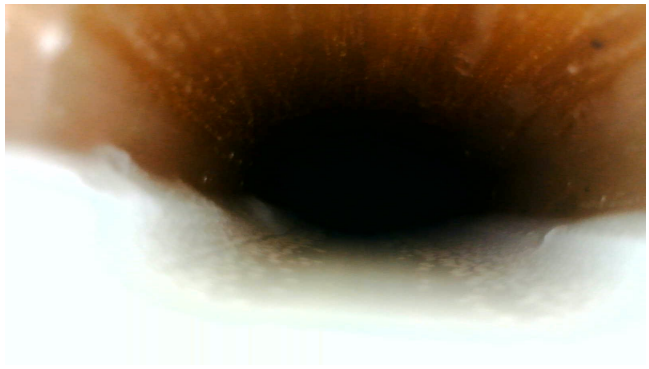

Figure 7.20: Picture taken 15cm inside copper pipe, inlet side

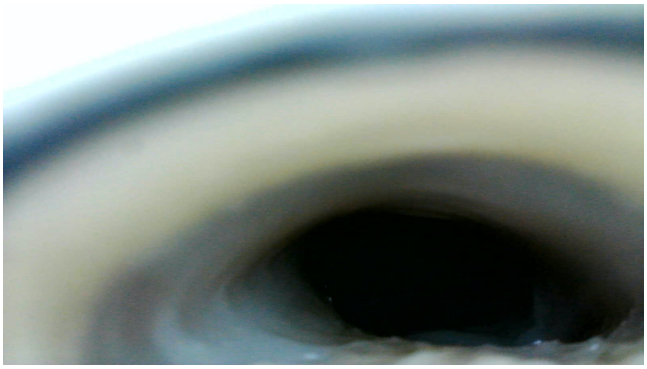

Figure 7.21: Picture taken 15cm inside copper pipe, outlet side

Run 8, Slug Flow $usl = 0.033$

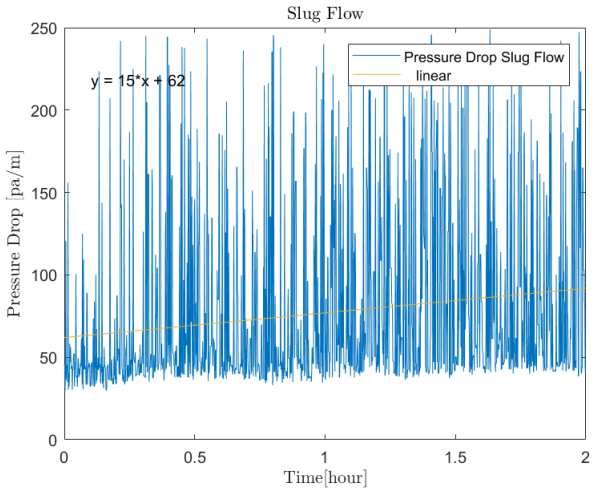

Figure 7.22: Pressure Drop increase over test section under Slug flow

Figure 7.23: Picture taken 15cm inside copper pipe, inlet side

Figure 7.24: Picture taken 15cm inside copper pipe, outlet side

Run 9, Stratified Flow $u_{sl} = 0.013$

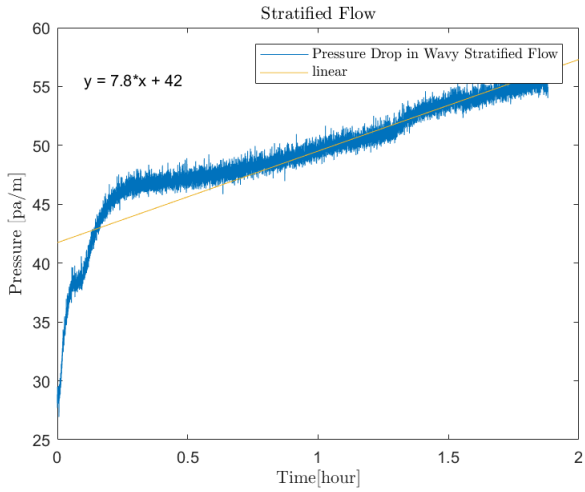

Figure 7.25: Pressure Drop increase over test section under stratified flow

Figure 7.26: Picture taken 15cm inside copper pipe, inlet side

Figure 7.27: Picture taken 15cm inside copper pipe, outlet side

Run 10, Stratified Flow $u_{sl} = 0.018$

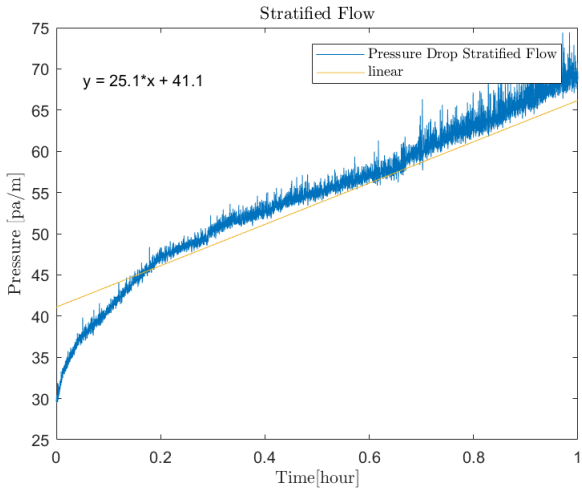

Figure 7.28: Pressure Drop increase over test section under Stratified flow

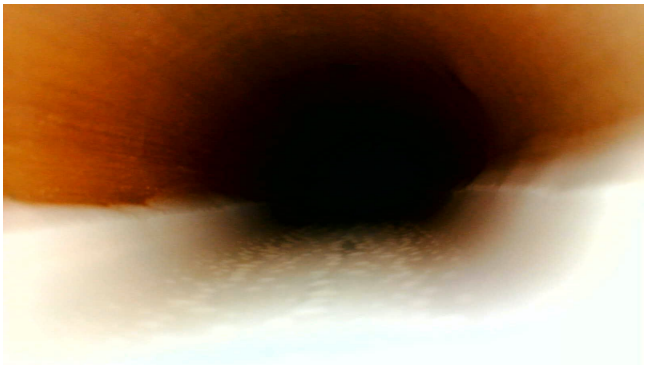

Figure 7.29: Picture taken 15cm inside copper pipe, inlet side

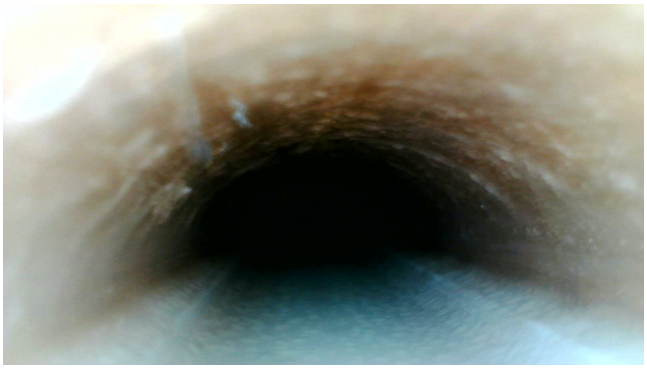

Figure 7.30: Picture taken 15cm inside copper pipe, outlet side

Run 11, Stratified Flow $u_{sl} = 0.021$

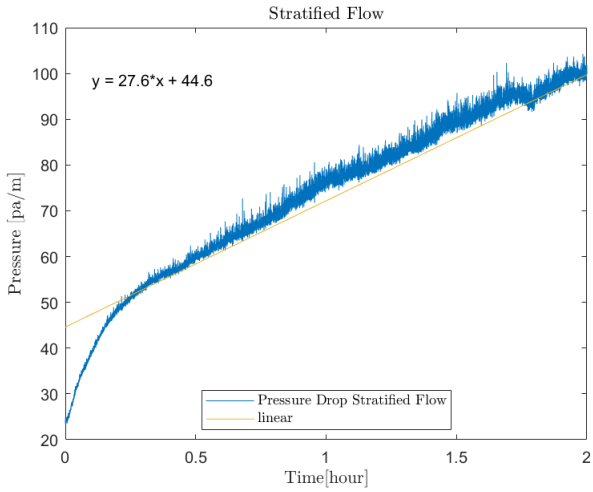

Figure 7.31: Pressure Drop increase over test section under stratified flow

Figure 7.32: Picture taken 15cm inside copper pipe, inlet side

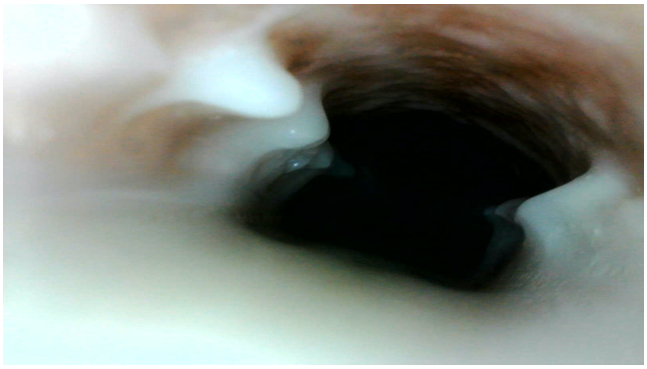

Figure 7.33: Picture taken 15cm inside copper pipe, outlet side

Run 12, Wavy Stratified Flow $u_{sl} = 0.028$

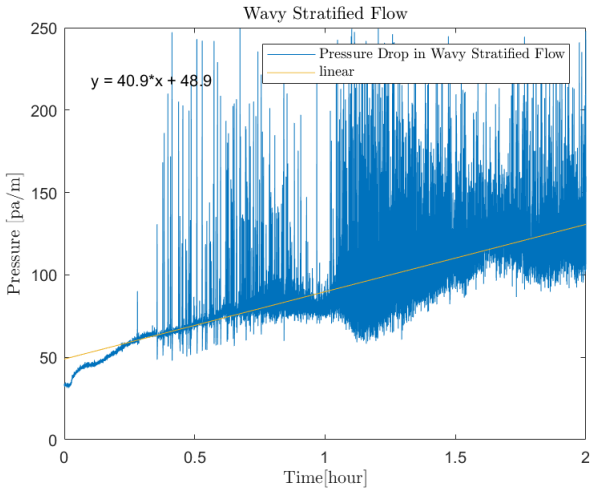

Figure 7.34: Pressure Drop increase over test section under Wavy Stratified flow

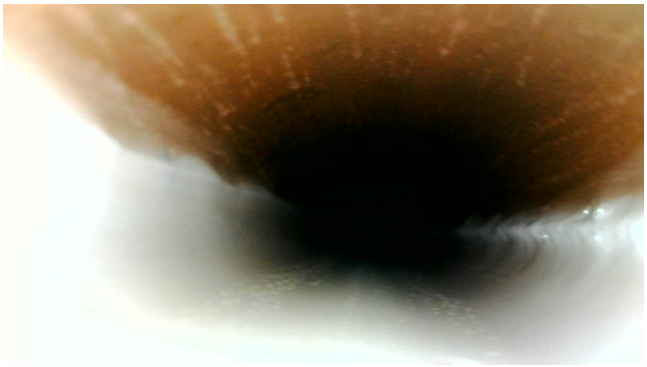

Figure 7.35: Picture taken 15 cm inside copper pipe, inlet side

Figure 7.36: Picture taken 15cm inside copper pipe, outlet side

Run 13, Stratified Flow $u_{sl} = 0.009$

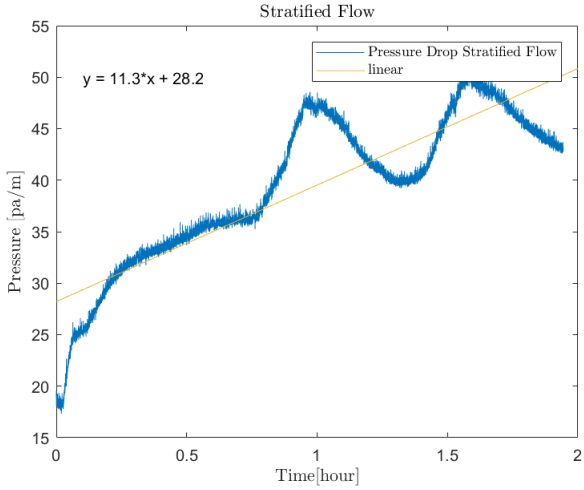

Figure 7.37: Pressure Drop increase over test section under Stratified Flow

Figure 7.38: Picture taken 15cm inside copper pipe, inlet side

Figure 7.39: Picture taken 15cm inside copper pipe, outlet side

Run 14, Wavy Stratified Flow $u_{sl} = 0.028$

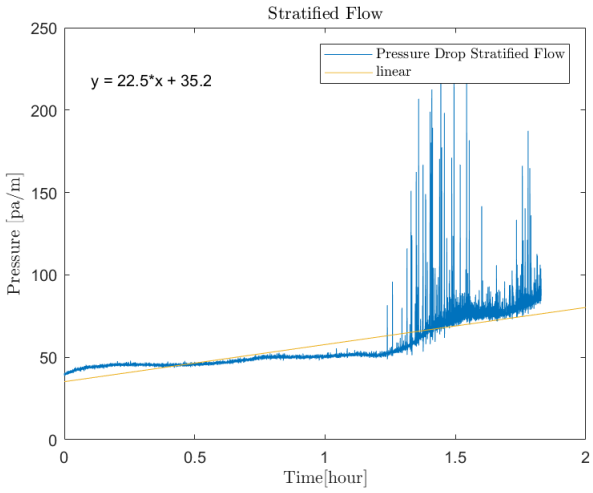

Figure 7.40: Pressure Drop increase over test section under Wavy Stratified Flow,

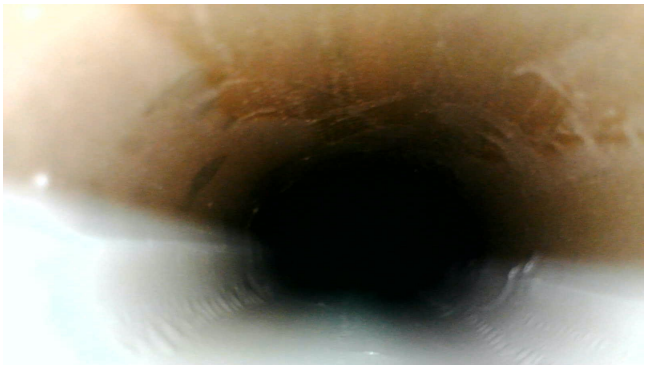

Figure 7.41: Picture taken 15cm inside copper pipe, inlet side

Figure 7.42: Picture taken 15cm inside copper pipe, outlet side

Run 15, Slug Flow $u_{sl} = 0.031$

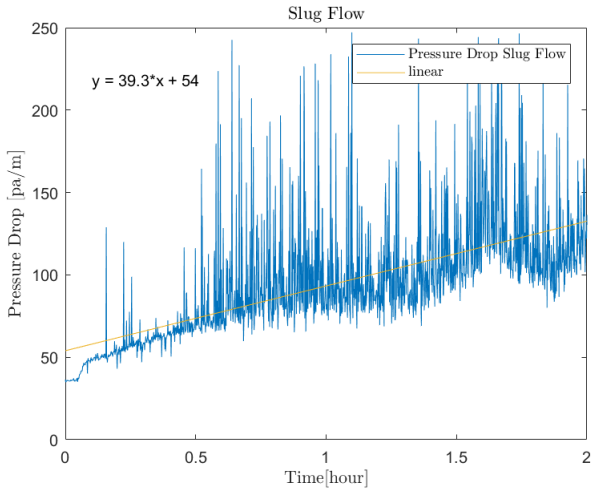

Figure 7.43: Pressure Drop increase over test section under Stratified Flow

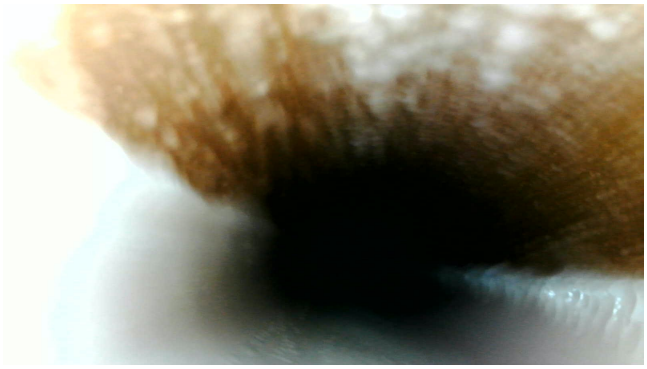

Figure 7.44: Picture taken 15cm inside copper pipe, inlet side

Figure 7.45: Picture taken 15cm inside copper pipe, outlet side

Run 16, Slug Flow $u_{sl} = 0.04$

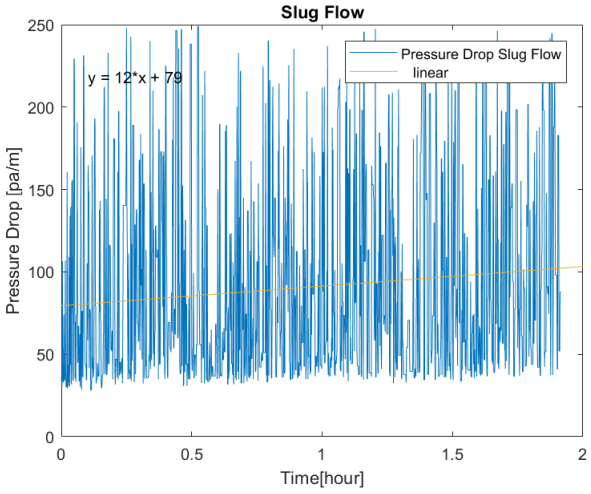

Figure 7.46: Pressure Drop increase over test section under slug flow

Figure 7.47: Picture taken 15cm inside copper pipe, inlet side

Figure 7.48: Picture taken 15cm inside copper pipe, outlet side

Run 17, Wavy Stratified Flow $u_{sl} = 0.026$

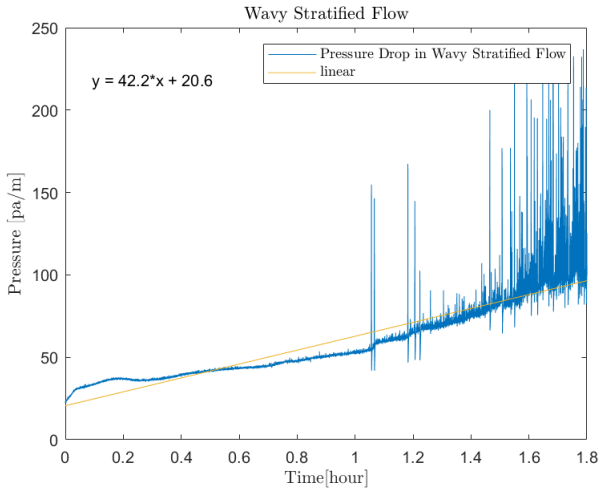

Figure 7.49: Pressure Drop increase over test section under Stratified Flow

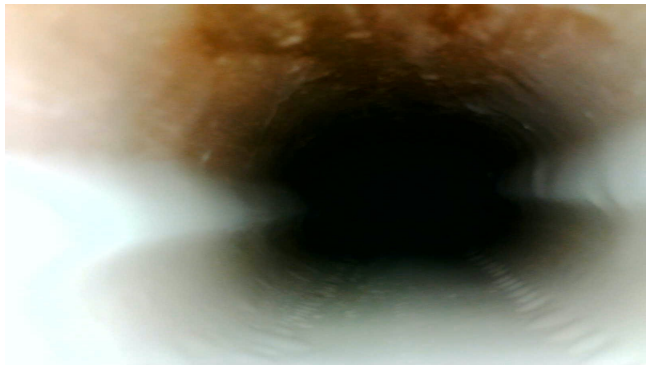

Figure 7.50: Picture taken 15cm inside copper pipe, inlet side

Figure 7.51: Picture taken 15cm inside copper pipe, outlet side

Appendix B

Risk Assessment Report

NTNU SINTEF

Risk Assessment Report

Wax Deposition Stand

Prosjektnavn	Wax Deposition in Multiphase Flow
Apparatur	Wax Deposition Stand
Enhet	NTNU-EPT
Apparaturansvarlig	Ole Jørgen Nydal
Prosjektleder	Ole Jørgen Nydal
HMS-koordinator	Morten Grønli
HMS-ansvarlig (linjeleder)	Terese Løvaas
Plassering	Basement
Romnummer	C082
Risikovurdering utført av	Yury Novoseltsev, Morten Grønli, Kristian Døble

Approval:

Apparatur kort (UNIT CARD) valid for:	12 months
Forsøk pågår kort (EXPERIMENT IN PROGRESS) valid for:	12 months

Rolle	Navn	Dato	Signatur
Prosjektleder	Ole Jørgen Nydal	27/4/18	
HMS koordinator	Morten Grønli		
HMS ansvarlig (linjeleder)	Terese Løvaas		

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Action on rig before evacuation:
The pumps should be turned off by emergency button on electrical cabinet. Cooler should be switched off by its own emergency button. Unplug all power electrical connections.

6 WARNING

6.1 Before experiments

Send an e-mail with information about the planned experiment to:
iept-experiments@ivt.ntnu.no

The e-mail must include the following information:

- Name of responsible person:
- Experimental setup/rig:
- Start Experiments: (date and time)
- Stop Experiments: (date and time)

You must get the approval back from the laboratory management before start up. All running experiments are notified in the activity calendar for the lab to be sure they are coordinated with other activity.

6.2 Non-conformance

FIRE
If you are NOT able to extinguish the fire, activate the nearest fire alarm and evacuate area. Be then available for fire brigade and building caretaker to detect fire place.
If possible, notify:

NTNU	SINTEF
Morten Grønli, Mob: 918 97 515	Linda Helander, Mob: +4740648621
Terese Løvaas: Mob: 918 97 209	Petter Røkke, Mob: +4790120221
NTNU – SINTEF Beredskapstelefon	800 80 388

GAS ALARM
If a gas alarm occurs, close gas bottles immediately and ventilate the area. If the level of the gas concentration does not decrease within a reasonable time, activate the fire alarm and evacuate the lab. Designated personnel or fire department checks the leak to determine whether it is possible to seal the leak and ventilate the area in a responsible manner.

PERSONAL INJURY

- First aid kit in the fire / first aid stations
- Shout for help
- Start life-saving first aid
- CALL 113 if there is any doubt whether there is a serious injury

OTHER NON-CONFORMANCE (AVVIK)

NTNU:
You will find the reporting form for non-conformance on:

1 INTRODUCTION

This experiment is used to find out wax deposition thickness depending of different cooling and multiphase flow conditions. Experimental stand is 3 m long featuring 2 m 1 inch test pipe in pipe with oil in inner pipe and cooling water in the annulus. Air is used for gas phase, Marcol 52 oil is used for liquid phase. Food grade wax is dissolved in the Marcol oil at temperatures less than 50 deg and then deposited in test pipe. Deposition is forced by cooling test section with water.

2 ORGANISATION

Rolle	
Prosjektleder	Ole Jørgen Nydal
Apparaturansvarlig	Ole Jørgen Nydal
Romansvarlig	Martin Bustadmo
HMS koordinator	Morten Grønli
HMS ansvarlig (linjeleder):	Terese Løvaas

3 RISK MANAGEMENT IN THE PROJECT

Hovedaktiviteter risikostyring	Nødvendige tiltak, dokumentasjon	DATE
Prosjekt initiering	Prosjekt initiering mal	
Veiledningsmøte Guidance Meeting	Skjema for Veiledningsmøte med pre-risikovurdering	
Innledende risikovurdering Initial Assessment	Fareidentifikasjon – HAZID Skjema grovanalyse	
Vurdering av teknisk sikkerhet Evaluation of technical security	Prosess-HAZOP Tekniske dokumentasjoner	
Vurdering av operasjonell sikkerhet Evaluation of operational safety	Prosedyre-HAZOP Opplæringsplan for operatører	
Sluttvurdering, kvalitetssikring Final assessment, quality assurance	Uavhengig kontroll Utstedelse av apparaturkort Utstedelse av forsøk pågår kort	

4 DESCRIPTIONS OF EXPERIMENTAL SETUP

See Attachments

5 EVACUATION FROM THE EXPERIMENTAL AREA

Evacuate at signal from the alarm system or local gas alarms with its own local alert with sound and light outside the room in question, see 6.2

Evacuation from the rigg area takes place through the marked emergency exits to the assembly point, (corner of Old Chemistry Kjelhuset or parking 1a-b.)

Conclusion:

7.6 Chemicals

YES	Marcol 52 Sasolwax 5603

Attachments: Data sheet of Marcol 52, Sasolwax 5603
Conclusion: Marcol 52 contains highly refined base oil and is not considered to present any hazard during normal use. Sasolwax 5603 contains highly refined paraffinic wax and is not considered to present any hazard during normal use.

7.7 Electricity safety (deviations from the norms/standards)

NO	

Attachments:
Conclusion:

8 ASSESSMENT OF OPERATIONAL SAFETY

Ensure that the procedures cover all identified risk factors that must be taken care of. Ensure that the operators and technical performance have sufficient expertise.

8.1 Procedure HAZOP

Attachments: HAZOP_MAL_ Procedure
Conclusion: Simplified procedure. Misunderstandings will not lead to unacceptable hazardous situations

8.2 Operation procedure and emergency shutdown procedure

Be careful to start the pumps slowly. It is vital that the step-vise start up and shut down procedure included in "HAZOP_MAL_ Procedure" is followed by the operators to avoid any severe damage to equipment and injury to the people.

Attachments: Procedure for running experiments
Emergency shutdown procedure:

- Turn off button on the electrical cabinet
- Turn off button on the K9 chiller
- Manually disconnect power plugs.
- Close air supply valve
- Now can leave in panic

8.3 Training of operators

The operators are responsible for running the tests in the wax deposition stand. They should have knowledge about procedures for the experiments, emergency shutdown, nearest fire and first aid station, and chemicals in the loop.

Attachments: Training of operators

https://innsida.ntnu.no/wiki/-/wiki/Norsk/Melde+avvik

SINTEF:
Synergi

7 ASSESSMENT OF TECHNICAL SAFETY

7.1 HAZOP

Node 1	Water Loop
Node 2	Air and oil Loop

Attachments: skjema: Hazop_mal
Conclusion: Working with the facility is simple and does not cause any serious problem. Other problems can be prevented by the emergency shut down.

7.2 Flammable, reactive and pressurized substances and gas

Are any flammable, reactive and pressurized substances and gases in use?

YES	Marcol 52 has a flash point > 148°C Pure paraffin wax SasolWax5603

Attachments: Data sheet of Marcol 52, Datasheet for SasolWax5603

7.3 Pressurized equipment

Is any pressurized equipment in use?

YES	Air buffer tank rated to 11 bar, acrylic pipes rated to 3 bar, flex hoses rated to 6 bar

Attachments:
Conclusion: As the outlet will be against atmospheric pressure and no clogging of the pipe is possible the pressure inside the stand should never exceed 2bars.

7.4 Effects on the environment (emissions, noise, temperature, vibration, smell)

Will the experiments generate emission of smoke, gas, odour or unusual waste?
Is there a need for a discharge permit, extraordinary measures?

YES	Flow leakage from the set-up will make the ground slippery. Exhaust air might carry some liquid component.

Conclusion: The set-up will be test checked with water for leaks. Be careful not to dispose any oil containing fluid in to the drains. They should be stored in the available barrels. Exhaust air will go through removal of any carried liquid, so it will be clean before release to environment.

7.5 Radiation

NO	

Attachments:

	Svært liten	A1	A2	A3	A4	A5
		Svært liten	Liten	Middels	Stor	Svært Stor
		PROBABILITY				

The principle of the acceptance criterion. Explanation of the colors used in the matrix

Colour		Description
Red		Unacceptable risk Action has to be taken to reduce risk
Yellow		Assessment area. Actions has to be considered
Green		Acceptable risk. Action can be taken based on other criteria

8.4 Technical modifications

The operators only allowed replacing broken parts with new parts similar to the old one. Modifications involving major changes to pressure characteristics of the system should only be done by lab technicians

8.5 Personal protective equipment

- Use of eye protection in the rig zone is mandatory.

8.6 General Safety

- The area around the rig should be cleaned. Buckets of oil/water should not be left by the rig.
- Operators cannot leave the Lab during experiments.

8.7 Safety equipment

Equipment for cleaning and removing of oil and water spill.

8.8 Special predations

9 QUANTIFYING OF RISK - RISK MATRIX

The risk matrix will provide visualization and an overview of activity risks so that management and users get the most complete picture of risk factors.

Idnr	Dangerous situation	Probability	Consequences	Combination
1	Oil-water spill: *Slippery floor. Oil/water kept in open containers*	3	B	B3
2	*Eye damage*	2	D	D2
3	*Electrocution (due to water spillage on motor)*	3	C	D2
4	*Loose cables and components around the rig. (Stumbling)*	3	B	B3
5	*Heat damage. (hot oil/ tank)*	2	B	C2

Conclusion: Probability of eye damage is small but consequences are dangerous, so, the use of eye protection glasses is mandatory. The ground around the rig should be dry and clean always. Routines for removal of oil/water spillage have to be established. Electrical connections to be shielded from water in reasonable way, motor elevated above the floor.

CONSEQUENSES						
	Svært alvorlig	E1	E2	E3	E4	E5
	Alvorlig	D1	D2	D3	D4	D5
	Moderat	C1	C2	C3	C4	C5
	Liten	B1	B2	B3	B4	B5

NTNU SINTEF

Attachment to Risk Assessment report

Wax Deposition Stand

Prosjektnavn	Wax Deposition in Multiphase Flow
Apparatur	Wax Deposition Stand
Enhet	NTNU-EPT
Apparaturansvarlig	Ole Jørgen Nydal
Prosjektleder	Ole Jørgen Nydal
HMS-koordinator	Morten Grønli
HMS-ansvarlig (linjeleder)	Olav Bolland
Plassering	Basement
Romnummer	C082
Risikovurdering utført av	Yury Novoseltsev, Morten Grønli, Kristian Døble

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ATTACHMENT A: PROCESS AND INSTRUMENTATION DIAGRAM

See the attached PDF for water loop schema and oil/air loop schema.

ATTACHMENT B: HAZOP TEMPLATE

Project: Wax Deposition Stand – Wax deposition experiments Node: 1 Water Loop						Page	
Ref	**Guideword**	**Causes**	**Consequences**	**Safeguards**	**Recommendations**	**Action**	**Date/Sign**
1.1	No flow	Pump in chiller not working. Operating at low pump frequency. Closed flow line valves. Damaged tubing	None	Physical inspection of pumps and valves. Ensure that the controls are functioning.	Stop pump. Safeguards in procedure.	Check for any pump damage. Check if all the valves are opened. Check tank level.	
1.2	Reverse flow	NA					
1.3	More flow	High pump frequency	Minor vibration of flow lines.	Operate within limits for flow rate. <13 l/min	Frequency limits in procedure	Check for any frequency converter issue.	
1.4	Less flow	Partial opening of the valves. Low pump frequency	Overloading of the pumps due to partial opening			Check for tank level and any frequency converter issue.	
1.5	More level	No air, fill the loop with water	None				
1.6	Less level	Inlet to pump closed or damaged	Pump running dry	Physical inspection of pumps inlet valves			
1.7	More pressure	Damaged tubing, valve not fully open	Safety valve releases the overpressure	Safety valve			
1.8	Less pressure	See 1.1					
1.9	More water	Ambient	Less cooling	Chiller in the area		Check	

Project: Wax Deposition Stand – Wax deposition experiments Node: 1 Water Loop						Page	
Ref	**Guideword**	**Causes**	**Consequences**	**Safeguards**	**Recommendations**	**Action**	**Date/Sign**
	temperature	temperature is high Heat exchanger not working properly	efficiency	with good ventilation. Self tests of the chiller		temperature and ventilation.	
1.10	Less temperature	NA					
1.11	More viscosity	NA					
1.12	Less viscosity	NA					
1.13	Composition Change	NA					
1.14	Contamination	NA					
1.15	Relief	N/A					
1.16	Instrumentation	Dirt, damage to sensors, wrong signal	No control of the system	Ensure the sensors are functioning		System will always also be visually monitored	
1.17	Sampling	NA					
1.18	Corrosion/erosion	NA					
1.19	Service failure	NA					
1.20	Abnormal Operation	NA					
1.21	Maintenance	NA					
1.22	Ignition	NA					
1.23	Spare equipment	NA					
1.24	Safety	Water/oil spill	Slippy floor	Physical inspection		Shutdown, clean, repair	

Project: Wax Deposition Stand – Wax deposition experiments Node: 2 Oil/Air Loop						Page	
Ref	**Guideword**	**Causes**	**Consequences**	**Safeguards**	**Recommendations**	**Action**	**Date/Sign**
2.13	Composition Change	Deposition on walls before test section	No hazardous consequences	Heating tapes with PID controllers	Increase heating of pipe walls, increase insulation	Check if pipes properly heated and insulated	
2.14	Contamination	NA					
2.15	Relief	High air pressure from air central system	Discharge to the atmosphere. Loudly noise	Safety valves and pressure gauge Ear protection available at operator's residence.			
2.16	Instrumentation	NA					
2.17	Sampling	NA					
2.18	Corrosion/erosion	NA					
2.19	Service failure	NA					
2.20	Abnormal operation	NA					
2.21	Maintenance	NA					
2.22	Ignition	NA					
2.23	Spare equipment	NA					
2.24	Safety	Increasing the air pressure.	Noise from discharging air. Breaking the Acrylic pipe.	Ear protection available at operator's residence.	Physical inspection		

Project: Wax Deposition Stand – Wax deposition experiments Node: 2 Oil/Air Loop						Page	
Ref	**Guideword**	**Causes**	**Consequences**	**Safeguards**	**Recommendations**	**Action**	**Date/Sign**
2.1	No flow	Closed flow line valves. No air supply.	Ref: Node 1:1	Ensure that all the flow line valves are open.	Stop pump.	Check if all the valves are opened.	
2.2	Reverse flow	Air in oil loop	No hazardous consequences	Non return valves			
2.3	More flow	Ref : Node 2:7					
2.4	Less flow	Partial opening of the valves.	Reverse flow of water/oil. Filling the tubing up to control valve.	Ensure that all the flow line valves are open Operational safety.	Stop pump.	Check if all the valves are opened.	
2.5	More level	NA					
2.6	Less level	NA					
2.7	More pressure	Open the air valve suddenly. Pressure regulator defect or modified Oil pump frequency is incorrect	Noise, incorrect readings	Loop is open to atmospheric pressure	Ensure that exhaust is not blocked	Close the air valve. Stop the oil pump	
2.8	Less pressure						
2.9	More temperature	Incorrect settings of the heating elements	Damage to flex hoses, acrylic pipes	Thermocouples readings. Alarm if temperature is higher level	Temperature upper limit clearly marked in procedure	Stop heating	
2.10	Less temperature	NA					
2.11	More viscosity	NA					
2.12	Less viscosity	NA					

ATTACHMENT C: TEST CERTIFICATE FOR LOCAL PRESSURE TESTING

Trykkpåkjent utstyr:	
Benyttes i rigg:	
Design trykk for utstyr (bara):	
Maksimum tillatt trykk (bara): (i.e. burst pressure om kjent)	
Maksimum driftstrykk i denne rigg:	

Prøvetrykket skal fastlegges i følge standarden og med hensyn til maksimum tillatt trykk.

Prøvetrykk (bara):			
X maksimum driftstrykk: I følge standard			
Test medium:			
Temperatur (°C)			
Start tid:		Trykk (bara):	
Slutt tid:		Trykk (bara):	
Maksimum driftstrykk i denne rigg:			

Eventuelle repetisjoner fra atm. trykk til maksimum prøvetrykk:.....

Test trykket, dato for testing og maksimum tillatt driftstrykk skal markers på (skilt eller innslått)

Sted og dato

Signatur

ATTACHMENT D: HAZOP PROCEDURE (TEMPLATE)

Project: Node: 1						Page	
Ref#	**Guideword**	**Causes**	**Consequences**	**Safeguards**	**Recommendations**	**Action**	**Date/Sign**
	Not clear procedure	Procedure is to ambitious, or confusingly					
	Step in the wrong place	The procedure can lead to actions done in the wrong pattern or sequence					
	Wrong actions	Procedure improperly specified					
	Incorrect information	Information provided in advance of the specified action is wrong					
	Step missing	Missing step, or step requires too much of operator					
	Step unsucessful	Step has a high probability of failure					
	Influence and effects from other	Procedure's performance can be affected by other sources					

ATTACHMENT E: PROCEDURE FOR RUNNING EXPERIMENTS

Prosjekt Wax deposition experiments	**Dato**	**Signatur**
Apparatur Wax Deposition Stand		
Prosjektleder Ole Jørgen Nydal	27/4/18	

	Conditions for the experiment:	**Completed**
	Experiments should be run in normal working hours, 08:00-16:00 during winter time and 08.00-15.00 during summer time. Experiments outside normal working hours shall be approved.	
	One person must always be present while running experiments, and should be approved as an experimental leader.	
	An early warning is given according to the lab rules, and accepted by authorized personnel.	
	Be sure that everyone taking part of the experiment is wearing the necessary protecting equipment and is aware of the shut down procedure and escape routes.	
	Preparations	**Carried out**
	Post the "Experiment in progress" sign.	
	Clear the area around the loop. There should be no open containers with oil/water or oil/water on the floor.	
	Put chiller outside the room for better ventilation	
	Put rubber mats above chiller hoses to ensure unobstructed escape route.	
	Check that exhaust pipe to atmosphere is going properly through water bath, so air is cooled and any oil carried by air is caught in the bath	
	Connect chiller, electrical cabinet, PID regulators to power supply	
	Open/close required valves for only air flow.	
	Run only air to check that air can freely escape to atmosphere by slowly opening air control valve	
	Open/close required valves for cooling water only flow	
	Run Water cooling for checking leakage/blockage.	
	Open/close required valves for oil only flow	
	Run cold oil flow for checking leakage/blockage	
	Start heating	
	Run hot oil without air or water cooling to clean deposited wax if required	
	During the experiment	
	Control of leakage in the set-up	
	Control air pressure, control oil tank temperature(alarm will help if temperature is exceeded max value), control cooling water temperature	
	End of experiment	
	Stop water cooling loop	
	Switch off air	
	Wait while hot oil removes wax deposited on the copper tube	

	By gradually increasing air content and decreasing oil content purge the system of oil	
	Turn off heating	
	Turn off the liquid pumps and close the control valves.	
	Then slowly close of the air control valve.	
	Valves should be back at the initial positions. Including main supply air feed valve.	
	Disconnect all electrical power supply	
	Put chiller back into the room	
	Remove all obstructions/barriers/signs around the experiment.	
	Tidy up and return all tools and equipment.	

Operator(s):

Navn	Dato	Signatur
Kristian Døble		

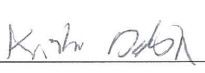
ATTACHMENT F: TRAINING OF OPERATORS

Prosjekt Wax Deposition Experiments	**Dato**	**Signatur**
Apparatur Wax Deposition Stand		
Prosjektleder Ole Jørgen Nydal		

	Knowledge to EPT LAB in general	
	Lab • Access • routines and rules • working hours	
	Knowledge about the evacuation procedures.	
	Activity calendar for the Lab	
	Early warning, iept-experiments@ivt.ntnu.no	
	Knowledge to the experiments	
	Procedures for the experiments	
	Emergency shutdown.	
	Nearest fire and first aid station.	
	Knowledge about the fluid in the loop • Marcol 52 • SasolWax 5603	
	Practical training to run the experiment	

I hereby declare that I have read and understood the regulatory requirements has received appropriate training to run this experiment and are aware of my personal responsibility by working in EPT laboratories.

Operator(s):

Navn	Dato	Signatur
Yury Novoseltsev		
Kristian Døble	27/04-2018	

ATTACHMENT G: FORM FOR SAFE JOB ANALYSIS

SJA name:	
Date:	Location:
Mark for completed checklist:	

Participators:		
SJA-responsible:		

Specification of work (What and how?):
Risks associated with the work:
Safeguards: (plan for actions, see next page):
Conclusions/comments:

Recommended/approved	**Date/Signature:**	**Recommended/approved**	**Date/Signature:**
SJA-responsible:		HSE responsible:	
Responsible for work:		Other, (position):	

HSE aspect	Yes	No	NA	Comments / actions	Resp.
Documentation, experience, qualifications					
Known operation or work?					
Knowledge of experiences / incidents from similar operations?					
Necessary personnel?					
Communication and coordinating					
Potential conflicts with other operations?					
Handling of an eventually incident (alarm, evacuation)?					
Need for extra assistance / watch?					
Working area					
Unusual working position					
Work in tanks, manhole?					
Work in ditch, shaft or pit?					
Clean and tidy?					
Protective equipment beyond the personal?					
Weather, wind, visibility, lighting, ventilation?					
Usage of scaffolding/lifts/belts/ straps, anti-falling device?					
Work at hights?					
Ionizing radiation?					
Influence of escape routes?					
Chemical hazards					
Usage of hazardous/toxic/corrosive chemicals?					
Usage of flammable or explosive chemicals?					
Risk assessment of usage?					
Biological materials/substances?					
Dust/asbestos/dust from insulation?					
Mechanical hazards					
Stability/strength/tension?					
Crush/clamp/cut/hit?					
Dust/pressure/temperature?					
Handling of waste disposal?					
Need of special tools?					
Electrical hazards					
Current/Voltage/over 1000V?					
Current surge, short circuit?					
Loss of current supply?					
Area					
Need for inspection?					
Marking/system of signs/rope off?					
Environmental consequences?					
Key physical security systems					
Work on or demounting of safety systems?					
Other					

NTNU SINTEF

APPARATURKORT / UNITCARD

Dette kortet SKAL henges godt synlig på apparaturen!

This card MUST be posted on a visible place on the unit!

Apparatur (Unit) Wax Deposition Stand	
Prosjektleder (Project Leader) Ole Jørgen Nydal	**Telefon mobil/privat** (Phone no. mobile/private)
Apparaturansvarlig (Unit Responsible) Ole Jørgen Nydal	**Telefon mobil/privat** (Phone no. mobile/private)
Sikkerhetsrisikoer (Safety hazards) Pressurized oil-water-air loop, leaks can give a dangerous jet for eyes. Chemicals used: Marcol 52, SasolWax 5603.	
Sikkerhetsregler (Safety rules) Safety goggles in the loop area. Gloves when handling the oil/chemicals	
Nødstopp prosedyre (Emergency shutdown) The pumps should be turned off by emergency switch on electrical cabinet; emergency button on chiller, power supply should be disconnected, air valve should be closed.	

Her finner du (Here you will find):

Prosedyrer (Procedures)	By the control desk
Bruksanvisning (Users manual)	By the control desk

Nærmeste (Nearest)

Brannslukningsapparat (fire extinguisher)	On the wall opposite to the rig
Førstehjelpsskap (first aid cabinet)	On the wall, left of the rig

NTNU
Institutt for energi og prosessteknikk

Dato

_____

Signert

_____

NTNU SINTEF

FORSØK PÅGÅR /EXPERIMENT IN PROGRESS

Dette kortet SKAL henges opp før forsøk kan starte!
This card MUST be posted on the unit before the experiment startup!

Apparatur (Unit) Wax Deposition Stand	
Prosjektleder (Project Leader) Ole Jørgen Nydal	**Telefon mobil/privat** (Phone no. mobile/private)
Apparaturansvarlig (Unit Responsible) Ole Jørgen Nydal	**Telefon mobil/privat** (Phone no. mobile/private)
Godkjente operatører (Approved Operators) Kristian Døble	**Telefon mobil/privat** (Phone no. mobile/private) 98636676
Prosjekt (Project) Wax Deposition Experiment	
Forsøkstid / Experimental time (start - stop)	
Kort beskrivelse av forsøket og relaterte farer (Short description of the experiment and related hazards**)** air-waxy oil flow in main loop with water flow in cooling loop Pressurized liquids, leaks can give a dangerous jet for eyes. Chemicals used: Marcol 52 oil, Sasolwax 5603 dissolved in oil Oil is hot max temperature 60 deg C	

NTNU
Institutt for energi og prosessteknikk

Dato

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Signert

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