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ONLINE MONITORING AND ANALYSIS OF WATER QUALITY IN OFFSHORE OIL & GAS PRODUCTION

BY
DENNIS SEVERIN HANSEN

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DENMARK

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Ph.D. Dissertation
Dennis Severin Hansen

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"Wonder is the beginning of wisdom."

— SOCRATES

Abstract

70% of the world's oil production derives from matured hydrocarbon fields. Water is together with oil trapped in the reservoirs and becomes a byproduct of the oil production, which steadily increases over time. In the Danish sector alone, the oil to water ratio (water cut) reached 80% in 2015, and the required amount of energy to treat the volumes of produced water before ocean discharge to comply with legislation increases. Another solution is to reinject the produced water into the reservoir to gain more sustainable production and comply with discharge legislation. Produced water reinjection has gained increasing attention in the last decades as it can increase yield and reduce oil discharge. However, the presence of solid particles, crude oil, and chemicals in the produced water, when reinjecting into the reservoir, introduces other complications in the injection water treatment process and the reservoir. Ideally, injection water should be sterile, non-scaling, free of suspended solids and oil to prevent plugging the reservoir rock's pores and reduce permeability. Furthermore, corrosion due to the presence of dissolved gases and microbially influenced corrosion bacteria should be prevented. A treatment process for achieving these ideal conditions is challenged to yield economic payoffs. The current solutions for measuring water quality are based on manual sampling, which suffers from being time-consuming and may not suffice to retain a high and consistent injection water quality. Despite the long history of implementing online quality monitors for measuring particle and oil droplet concentrations and sizes, there is no consensus regarding which method provides the most reliable estimate leaving petroleum engineers with an array of choices.

An extensive review study has been carried out of how different water quality issues can add to the suspended solids concentration that can potentially cause injectivity decline in the injection water treatment facility or, even worse, cause formation damage. Furthermore, the complexity of selecting quality monitors that reliably measure the concentration and sizes of suspended solids in the process has been heavily reviewed. Through the review process, online microscopy analyzers were deemed the best candidates for measuring single-particle properties within an injection water treatment process. Two different microscopy analyzers for measuring suspended particles have been validated,

including four identical fluorescence-based monitors for measuring oil-in-water concentrations. For each monitor type, a thorough calibration validation has been proposed to investigate their accuracy and reliability. The four identical fluorescence-based monitors for measuring oil-in-water concentrations revealed that the calibration procedure using a weighted least square yields higher reproducibility compared to ordinary least square due to the heteroskedastic behavior. The experimental validation results of the four fluorescence-based monitors showed a high precision between each other. However, due to its high sensitivity to fluorescent substances (fluorophores), they will be challenged in a dynamic separation facility with continuous changes of the fluorophores. The two different types of microscopy analyzers showed promising results in estimating known particle sizes and were able to measure oil-in-water concentrations with high precision in steady-state. A trailing moving average window of 1min was proposed resulting as a feasible solution for continuous measurements. It is acknowledged that an issue will arrive at shallow concentrations as the required number of oil droplets will not be captured within the expected relative error to represent the size distribution. Additionally, the classification procedure for both microscopy analyzers needs further validation.

Lastly, a connection between an industrial human-machine interface software and an academic used software were established to reduce the gap between academic research and industrial implementation. The connection was successfully established, and a case study of a model-predictive control method in the industrial used software was presented. The industrial human-machine interface software connection is a beneficial tool when, i.e., the presentation of water quality should be furtherly investigated.

Resumé

70% af verdens olieproduktion stammer fra aldrende oliefelter. Vand er sammen med olie fanget i reservoirerne og er derfor et biprodukt af olieproduktionen. Grundet udtømningen af oliefelterne stiger biproduktet af produceret vand støt hvert år. Alene i den danske sektor nåede olie/vand-forholdet 1/5 i 2015. Den nødvendige mængde energi til at behandle det producerede vand før udledning til havet stiger løbende, for at overholde udledningslovgivningen. En anden løsning er at geninjicere det producerede vand ned i reservoiret for at opretholde en mere bæredygtig produktion og overholde udledningslovgivningen. Injektion af produceret vand har fået øget opmærksomhed i de sidste årtier, da det kan øge det totale udbytte og reducere olieudledningen. Dog bliver behandlingsprocessen mere kompliceret når det producerede vand introduceres til injektionsprocessen, hvor tilstedeværelsen af faste partikler, råolie og kemikalier skal filtreres og behandles før det injiceres ned i reservoiret. Idelt set skal injektionsvandet være sterilt, fri for krystallisering, faste partikler og olie for at forhindre tilstopning af den interne porestruktur i reservoiret, og derved reducere permeabiliteten. Derudover bør gaskorrosion og mikrobiel korrosion forhindres. Men økonomisk set vil det være svært at opnå profit af olieproduktionen, hvis der tilstræbes en ideel behandlingsproces.

De nuværende målemetoder af vandkvalitet er baseret på manuelle prøvetagninger, som kan være tidskrævende, og vil derfor ikke være tilstrækkelig til at opretholde en høj og ensartet kvalitet af injektionsvandet. På trods af mange års implementering af forskellige typer af online enheder til måling af partikelstørrelser og koncentrationer er der ingen enighed om hvilken metode der giver den mest pålidelige måling, hvilket resulterer i at olieingeniører står tilbage med et svært valg når der skal investeres i nye måleenheder.

En gennemgribende undersøgelse er blevet udført omkring hvilke forskellige partikulære stoffer der kan være til stede i injektionsvandet, og dermed øge den samlede koncentration af suspenderede stoffer. De suspenderede stoffer kan potentielt forårsage reduktion af den injicerede vandmængde i injektionsprocessen, eller forårsage formationsskader i reservoiret. Derudover er kompleksiteten ved at vælge den rette type af måleenhed til at måle koncentrationer og partikelstørrelser af suspenderede stoffer i injektionsprocessen blevet undersøgt. Ud

fra analysen blev online mikroskopienheder konkluderet til at være de bedste kandidater til måling af forskellig partiklers fysiske egenskaber i en injektions-proces.

To forskellige online mikroskopienheder til måling af suspenderede partikler er blevet testet. Herudover er fire identiske fluorescensbaserede enheder til måling af olie-i-vand koncentrationer også blevet testet. For hver af de to enhedstyper er der givet en grundig kalibreringsvalidering for at undersøge deres nøjagtighed og pålidelighed. De fire identiske fluorescensbaserede enheder til måling af olie-i-vand koncentrationer eftervist at kalibreringsproceduren med anvendelse af en vægtet mindste kvadraters metode, giver øget reproducerbarhed sammenlignet med almindelig mindste kvadraters metode på grund af den heteroskedastiske opførsel af prøvetagningen under kalibreringen. De eksperimentelle valideringsresultater for de fire fluorescensbaserede enheder viste høj præcision mellem hinanden. Men på baggrund af deres høje følsomhed over for fluorescerende stoffer (fluoroforer) vil de blive udfordret i et dynamisk separationsanlæg med kontinuerlige ændringer af fluoroforer.

De to forskellige typer mikroskopienheder viste lovende resultater ved estimering af kendte partikelstørrelser, og var i stand til at måle olie-i-vand koncentrationer med høj præcision i stationær tilstand. Et glidende gennemsnitsvindue på 1min blev påvist som en mulig løsning til at måle koncentrationen kontinuerligt. Det erkendes dog at et af problemerne ved målinger af partikler og koncentrationer med online mikroskopienheder kan opstå, hvis et given antallet af partikler ikke opfanges inden for en acceptabel usikkerhed af størrelsesfordelingen. Klassificeringsproceduren for begge mikroskopienheder kræver yderligere validering.

Til sidst blev der etableret en forbindelse mellem en industriel brugerflade (HMI) og et akademisk software for at reducere afstanden mellem akademisk forskning og industriel implementering. Forbindelsen blev succesfuldt oprettet, hvilket er præsenteret i et casestudie med en demonstration af en model forudsigelig kontrol (MPC) metode på i industrielle brugerflade. Den industrielle brugerflade er et nyttigt værktøj når præsentationen af vandkvaliteten bør undersøges yderligere.

Contents

Thesis Details

Thesis Title: Online Monitoring and Analysis of Water Quality in
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Ph.D. Student: Dennis Severin Hansen
Supervisor: Associate Prof. Zhenyu Yang, Aalborg University, DK

The body of the thesis consists of the following papers:

[**A**]   D. S. Hansen, M. V. Bram, S. M. Ø. Lauridsen, and Z. Yang, "Online
          Quality Measurements of Total Suspended Solids for Offshore Rein-
          jection: A Review Study" *Energies*, MDPI, p. 53, 2020. [submitted
          for publication]

[**B**]   D. S. Hansen, M. V. Bram, and Z. Yang, "Efficiency investigation of
          an offshore deoiling hydrocyclone using real-time fluorescence- and
          microscopy-based monitors," in *2017 IEEE Conference on Control
          Technology and Applications (CCTA)*. Mauna Lani, HI: IEEE, 27-30
          Aug. 2017, pp. 1104–1109. Doi: 10.1109/CCTA.2017.8062606

[**C**]   D. S. Hansen, S. Jespersen, M. V. Bram, and Z. Yang, "Uncertainty
          Analysis of Fluorescence-Based Oil-In-Water Monitors for Oil and
          Gas Produced Water," *Sensors*, vol. 20, no. 16, p. 36, 2020. Doi:
          10.3390/s20164435

[**D**]   D. S. Hansen, S. Jespersen, M. V. Bram, and Z. Yang, "Offshore
          Online Measurements of Total Suspended Solids using Microscopy
          Analyzers" *IEEE Sensors Journal*, IEEE, p. 10, 2020. [submitted
          for publication]

[**E**]   D. S. Hansen, S. Jespersen, M. V. Bram, and Z. Yang, "Human
          Machine Interface Prototyping and Application for Advanced Con-
          trol of Offshore Topside Separation Processes," in *IECON 2018 -
          44th Annual Conference of the IEEE Industrial Electronics Society*.
          Washington, DC: IEEE, 21-23 Oct. 2018, pp. 2341–2347. Doi:
          10.1109/IECON.2018.8591309

In addition the following publications have also been made:

[1] P. Durdevic, S. Pedersen, M. Bram, D. Hansen, A. Hassan, and Z. Yang, "Control Oriented Modeling of a De-oiling Hydrocyclone," *IFAC-PapersOnLine*, vol. 48, no. 28, pp. 291–296, 2015. Doi: 10.1016/j.ifacol.2015.12.141

[2] M. V. Bram, A. A. Hassan, D. S. Hansen, P. Durdevic, S. Pedersen, and Z. Yang, "Experimental modeling of a deoiling hydrocyclone system," in *2015 20th International Conference on Methods and Models in Automation and Robotics (MMAR)*. Miedzyzdroje: IEEE, 24-27 Aug. 2015, pp. 1080-1085, Doi: 10.1109/MMAR.2015.7284029

[3] D. Hansen, M. Bram, P. Durdevic, S. Jespersen, and Z. Yang, "Efficiency evaluation of offshore deoiling applications utilizing real-time oil-in-water monitors," in *Oceans 2017 - Anchorage*. Anchorage, AK: IEEE, 18-21 Sep. 2017, p. 6. Available: https://ieeexplore.ieee.org/document/8232154

[4] P. Durdevic, C. Raju, M. Bram, D. Hansen, and Z. Yang, "Dynamic Oil-in-Water Concentration Acquisition on a Pilot-Scaled Offshore Water-Oil Separation Facility," *Sensors*, vol. 17, no. 124, p. 11, 2017. Doi: 10.3390/s17010124

[5] M. V. Bram, L. Hansen, D. S. Hansen, and Z. Yang, "Grey-Box Modeling of an Offshore Deoiling Hydrocyclone System," in *2017 IEEE Conference on Control Technology and Applications (CCTA)*. Mauna Lani, HI: IEEE, 27-30 Aug. 2017, pp. 94–98. Doi: 10.1109/CCTA.2017.8062446

[6] P. Andreussi, M. Bonizzi, P. Ciandri, S. Pedersen, and D. S. Hansen, "The use of a low-cost gas-liquid flow meter to monitor severe slugging," in *18th International Conference on Multiphase Production Technology*, Cannes: BHR Group, 7-9 Jun. 2017, pp. 413–424.

[7] M. V. Bram, L. Hansen, D. S. Hansen, and Z. Yang, "Hydrocyclone Separation Efficiency Modeled by Flow Resistances and Droplet Trajectories," *IFAC-PapersOnLine*, vol. 51, no. 8, pp. 132–137, 2018. Doi: 10.1016/j.ifacol.2018.06.367

[8] M. V. Bram, L. Hansen, D. S. Hansen, and Z. Yang, "Extended Grey-Box Modeling of Real-Time Hydrocyclone Separation Efficiency," in *2019 18th European Control Conference (ECC)*. Naples: IEEE, 25-28 Jun. 2019. pp. 3625–3631. Doi: 10.23919/ECC.2019.8796175

[9] M. V. Bram, S. Jespersen, D. S. Hansen, and Z. Yang, "Control-Oriented Modeling and Experimental Validation of a Deoiling Hydrocyclone System," *Processes*, vol. 8, no. 9, p. 33, 2020. Doi: 10.3390/pr8091010

[10] M. V. Bram, J. Calliess, S. Roberts, D. S. Hansen, Z. Yang, "Analysis and Modeling of State-Dependent Delay in Control Valves," In *IFAC World Congress 2020* (IFAC-PapersOnLine). Berlin: Elsevier, 11-17 Jul. 2020. [Accepted/In press]

[11] Z. Yang, P. Durdevic, S. Jespersen, and D. S. Hansen, "Potential of using Real-time OIW Monitoring for Control of Produced Water Treatment in Offshore Oil & Gas Production," *Advances in Science, Technology & Innovation*, Springer, 2020, p. 2. [Accepted/In press]

This thesis has been submitted for assessment in partial fulfillment of the Ph.D. degree. The thesis is based on the submitted or published scientific papers that are listed above. The thesis's body consists directly of the Papers A−E, where Papers 1−11 are additional papers related to offshore oil and gas. As part of the assessment, co-author statements have been made available to the assessment committee and available at the faculty.

Preface

This thesis is submitted as a collection of papers to fulfill the requirements for the degree of Doctor of Philosophy at the Department of Energy Technology, Aalborg University, Denmark. The entire work has been conducted at the Department of Energy Technology, Aalborg University, Denmark. The work was done in close collaboration with Total Denmark in the period September 2017 to December 2020 under the supervision of Associate Professor Zhenyu Yang. The project was supported by the Danish Hydrocarbon Research and Technology Centre and Aalborg University joint project: "Injection Water Quality and Control, Aalborg University, Pr-no: 878040". A direct thanks to the contact person at Total, Steven M. Ø. Lauridsen, for his support and collaboration during the project.

I gratefully thank all the colleagues from the Department of Energy Technology, Aalborg University, for providing a friendly research environment. I want to thank my supervisor Zhenyu Yang for his professional opinion on navigating in the academic research society and always finding time for discussions. Thanks to my project colleagues: Stefan Jespersen, Leif Hansen, Kasper Jepsen, Simon Pedersen, and Petar Durdevic, for providing great feedback, collaboration, and valuable discussions. Special thanks to my colleague, former fellow student, and friend, Mads V. Bram, for more than nine years of professional teamwork; it has been an honor. Lastly, I would like to express my gratitude to my girlfriend and my two kids, which has been part of my journey to hand in my Ph.D. thesis. They have been the foundation outside the research environment where I could relax.

<div align="right">

Dennis Severin Hansen
Aalborg University

</div>

Preface

Abbreviations

ASM	Abnormal Situation Management
CCD	Charge-coupled device
CMOS	Complementary metal–oxide–semiconductor
DS	Danish Standard
EIA	Energy Information Administration
ESV	Edge strength value
FRV	Focus rejection value
GC-FID	Gas chromatography-flame ionization detector
GHG	Greenhouse gases
HMI	Human machine interface
IEA	International Energy Agency
ISO	International Organization for Standardization
IW	Injection water
IWT	Injection water treatment
MIC	Microbially influenced corrosion
MPC	Model predictive control
OCED	Organisation for Economic Co-operation and Development
OiW	Oil-in-water
OLS	Ordinary least square
OPC	Open platform communications
OPEX	Operational expenditure
OSPAR	Oslo and Paris convention

P&ID	Piping and instrumentation diagram
PDR	Pressure different ratios
PID	Proportional, integral, and derivative
PLC	Programmable logic controller
PMT	Photomultiplier tube
ppm	Parts per million
PSD	Particle size distribution
PW	Produced water
PWRI	Produced water reinjection
RET	Resonance energy transfer
RFU	Relative fluorescence unit
SAE	Society of Automotive Engineers
SCADA	Supervisory control and data acquisition
TDS	Total dissolved solids
THV	Threshold value
TPES	Total primary energy supply
TSS	Total suspended solids
UV	Ultraviolet
WLS	Weighted least square

Nomenclature

$[c_0, c_1]$ Slope and the intercept on the ordinate axis for the predicted variance function

$[x, y]$ Cartesian coordinates

α non-zero exponential constant, for log-normal distribution $\alpha = 2$

$^{-}$ Arithmetic mean

β Basis number, for the count basis $\beta = 0$

δ Relative error

$\hat{}$ Prediction

μ Mean

μ_0 Mean of the log-normal distribution

ω Intermediate parameter

ω_i Weighting factor

σ Population Standard deviation

σ_0 Standard deviation of the log-normal distribution

$_0$ Set of observations

$_i$ Index observation from 1 to n

a Slope

b The intercept on the ordinate axis

c constant dependent on α and β

CI Confidence interval of linear regression curve

DOF Degree of freedom

e Error between predicted and observed data

k Coverage factor

n Number of observations

n^* Minimum number of required counted particles

$OiW_{repeat.}$ OiW concentration results with repeatability uncertainties from the reference method

$OiW_{reprod.}$	OiW concentration results with reproducibility uncertainties from the reference method
PI	Prediction interval of linear regression curve
s	Sample standard deviation
SS_{xx}	Weighted sum of square
SSE	Sum of square errors
$t_{0.025}$	t-score with two-tailed 95% confidence interval
u	Defined confidence level with its represented z-score value, for 95% confidence interval $u = 1.96$
X_N	Number of counted particles

Part I

Extended Summary

Chapter 1

Introduction and Motivation

1.1   Introduction

While the fossil fuel-based energy consumption in developed countries approaches a plateau and we are entering a new era of climate change, striving to reduce the carbon dioxide (CO_2) emission during the coming decades, production of oil is still expected to increase by 0.6% globally to meet the increasing demands the next 30 years [1, 2]. With an increasing population in developing countries and increasing energy demands, the Energy Information Administration (EIA) predicts that by 2050, natural gas, crude oil, and other liquids will still account for 48.4% of all combined energy sources, unless radical changes occur, as shown in Fig. 1.1 [2]. The total primary energy supply (TPES) in Fig. 1.1, refers to the amount of energy aggregate from production and imports without including exported energy.

   If the estimations that are shown in Fig. 1.1 are correct, oil will account for ~27% of the total energy consumption, hence the oil and gas industry will be a significant part of the worlds energy consumption during the next 30 years [4, 5]. Therefore, there is a strong incentive and a responsibility for the oil and gas industry to minimize greenhouse gases (GHG) emissions by investing in innovative solutions and exploring new technologies [6]. However, it must be taken into account that countries like Denmark, and many other developed European countries, have a goal of being CO_2 neutral within the next few decades. This is also shown in Fig. 1.1, as Organisation for Economic Co-operation and Development (OCED) countries are estimated to reduce the energy consumption from oil by 5.5$p.p.$, though OCED members represent a wide range of different countries around the world. Even if Denmark achieves to become CO_2 neutral in 2050, Denmark only accounts for 1‰ of the total

Access to Energy – Projection by EIA

2019	2030	2050
TPES: 662 Exajoules	TPES: 744 Exajoules	TPES: 961 Exajoules

2019

7.8 billion people
OCED: 1.3 billion people
Non-OCED: 6.4 billion people

1.0 billion people have no electricity*

2.7 billion people have no access to
clean fuels and technology for cooking*

2030

8.5 billion people
OCED: 1.4 billion people
Non-OCED: 7.2 billion people

0.7 billion people have no electricity*

2.3 billion people have no access to
clean fuels and technology for cooking*

2050

9.7 billion people
OCED: 1.4 billion people
Non-OCED: 8.2 billion people

0.3 billion people have no electricity**

1.7 billion people have no access to
clean fuels and technology for cooking**

Legend: Oil and other liquids · Natural gas · Other energy sources · OCED countries · Non-OCED countries

*Based on estimated data from the International Energy Agency (IEA) [3].
**Assuming a continuing gain of 3.34% and 1.40% annually for access to electricity and clean-cooking, respectively, based on the projected data from 2016 to 2030 by IEA [3].

Fig. 1.1: The presented data show the projection by EIA over a 31-years period [4]. Oil and other liquids include all types of crude oil, natural gas plant liquids, and biofuels. the other energy sources include coal, nuclear, and renewables, with renewables estimated to increase by an annually average of 3.1% worldwide. Thus, it is estimated to surpass energy consumption from oil and other liquids in 2047. Figure is from Paper A.

CO_2 emissions globally. Denmark's achievement of having a net-zero emission in 2050 must therefore be found in the positive return of being an inspiration for other countries to increase their ambition to reduce their CO_2 emissions. If this is not the case, pioneering countries such as Denmark should consider whether there is a risk of introducing "carbon leakage" instead, which will have a negative effect on the global CO_2 reduction [7]. Carbon leakage refers to the phenomenon of stricter GHG emissions regulations only causes production to increase in other countries with more lenient legislation [7]. That could be the case with the Danish oil and gas sector as they have the lowest carbon intensity from the upstream processes in the world [8]. The low carbon intensity is a positive outcome of a long history of stricter legislation and documentation regarding offshore production in the North Sea, governed by Oslo and Paris

convention (OSPAR) regulations and national legislation. One can, therefore, raise the question: "Can Denmark support disadvantageous actions to the environment by stopping the offshore oil production too soon through carbon leakage?"

In the transition to more sustainable production, and as the underground hydrocarbon reservoirs mature, offshore oil and gas production faces another difficult challenge; the increasing amount of produced water (PW). PW is the byproduct during the extraction of oil or gas. Producers in the Danish part of the North Sea have since 1986 injected water back into the underground reservoir to such an extent that the extracted mixture consists of more than 90% PW, as shown in Fig. 1.2 [9, 10]. In addition, the amount of oil produced from the reservoirs decreases over time, which has caused Denmark to no longer be a net exporter of oil as the Danish oil consumption exceeded the oil production in 2018 [11]. Therefore, it raises the second question: "How long can the operation remain financially feasible?" [12].
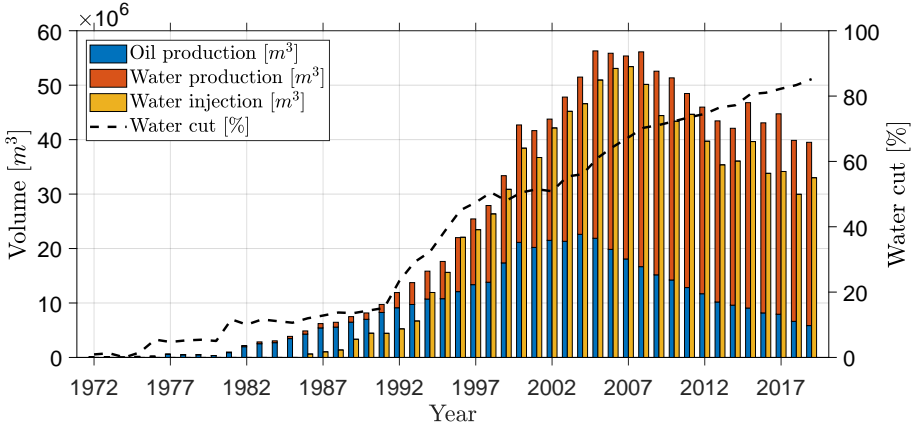
Fig. 1.2: The bar chart shows the annual production of oil and water, and amount of water that is injected in the Danish sector of the North Sea. The data are annually published by Danish Energy Agency [9]. Figure is from Paper A.

A general trend towards more sustainable production, guided by discharge legislation that introduces stricter legislation and strives to achieve zero discharge, improved enhanced recovery techniques, such as produced water reinjection (PWRI) have received increasing attention. The Danish part of the North Sea's current regulation states that oil-in-water (OiW) concentration must be less than 30ppm before discharging into the ocean. The Danish Environmental Protection Agency furthermore states that the total annual quantity of oil discharged into the North Sea must be less than 222t/yr based on an agreement from 2017/2018 [13–16]. As an increasing amount of water is pro-

duced, the limit of the total discharged oil becomes more significant, especially as the former Mærsk Oil in 2015 reached 96% of the allowed total amount of oil discharge [14, 15].

In 2014 the OSPAR commission reported that 16 installations failed to meet the 30ppm discharge legislation, where each of the installations exceeded an annual average concentration of dispersed oil discharge [17]. Several of the platforms reported that the reason for not achieving the standard was due to PWRI failure, which questions whether the OiW concentration in the PW is even considered when reinjecting. Increasing the PWRI ratio has the potential to increase yields and reduce oil emissions while complying with national and local regulations and minimizing negative environmental impacts [18–20]. The injection water (IW) is mainly injected to maintain reservoir pressure, which drives reservoir production. The secondary purpose is to sweep the reservoir, by displacing the oil to flow towards the production wells and thereby increase the oil production [21, 22].

In Denmark, $33.7 \cdot 10^6 \mathrm{m}^3$ of water was produced, and $33.0 \cdot 10^6 \mathrm{m}^3$ of water was injected during 2019, of which ∼14% of the IW consists of PW according to the Danish Environmental Protection Agency [23], while the remaining IW consists of treated seawater. To further increase the PWRI ratio, it must be economically feasible for acquiring sufficient quality to avoid accidental formation damage. Current offshore platforms rely on offline measurements of both OiW and total suspended solids (TSS) concentration, where OiW concentrations of PW must be measured manually at least twice daily. In comparison, reports of TSS concentrations from injection water treatment (IWT) facilities are not governed by legislation but governed by the producer's own choice. How often the TSS concentrations are measured can vary widely. Both manual reporting methods for OiW and TSS suffer from being time-consuming and may not suffice to increase the PWRI ratio. To increase the PWRI ratio, the IW quality must be high and consistent. Effective control of PW and IW involves appropriate treatment, discharge, and monitoring. Monitoring PW discharge can also help protect the receiving environment. Accurate water analysis is essential to gain an understanding of the dispersed conditions in PW and IW to identify changes in the process. Therefore, the operator must have confidence in the data that are obtained from the monitor. Accurate information on the amount of oil and particles, sizes, and classification of particles in IW can be used for decision support, reporting, or even advanced control to achieve better operation in the treatment process, all of which benefits water-intensive operation [20]. Therefore, the importance and awareness of accurate online water quality measurements have received more attention the last decades within the oil and gas industry. Based on a preliminary feasibility study, this thesis investigated two promising online monitor types: a fluorescence-based monitor for measuring the OiW concentration and two different microscopy analyzers for measuring particles. All three types of monitors are shown in Fig. 1.3.

(a)

(b)

(c)
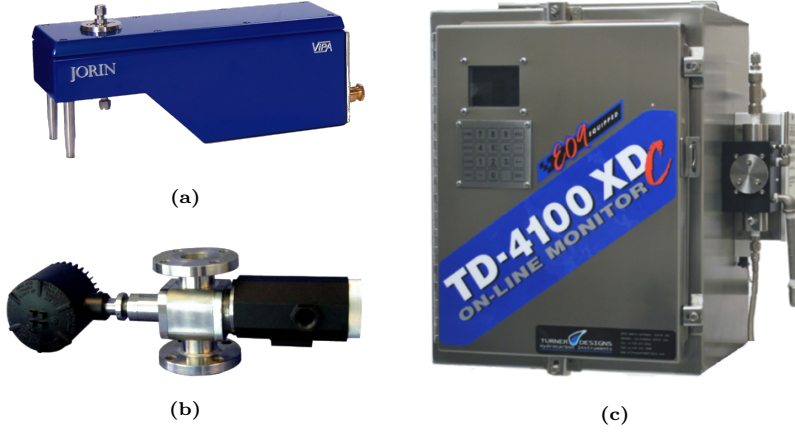
Fig. 1.3: The three types of online monitors used in the experiments described in this thesis: (**a**) Jorin ViPA; (**b**) Canty InFlow; (**c**) Turner TD-4100XDC.

1.1.1 Fluorescence-Based Monitor

The types of monitors described in this thesis, both microscopy and fluorescence spectroscopy, uses light in their measurement principles. Light is the smallest quantity of energy that can be transported. Light can be described in terms of a stream of photons traveling as a wave-particle duality at the speed of light [24]. A photon is an elementary particle that only can be created or destroyed. Looking at light in the electromagnetic spectrum, the visible light that the human eye is sensitive to only covers a very narrow range of the entire spectrum, as shown in Fig. 1.4 [25], from near-ultraviolet (UV) at a wavelength of 380nm to deep red at 710nm [25].
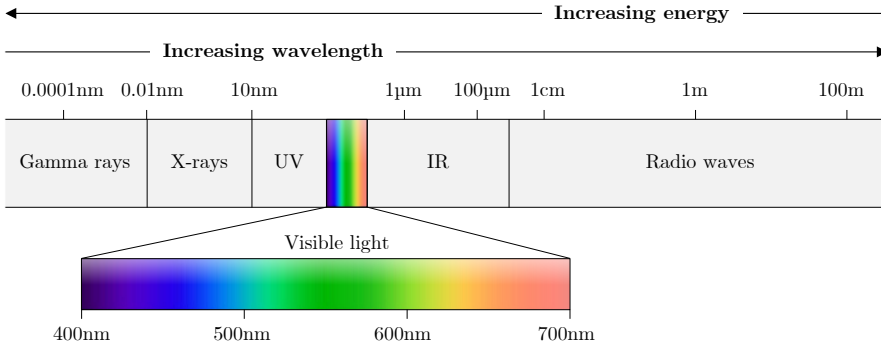
Fig. 1.4: The electromagnetic spectrum, with a zoom-in on the visible spectrum from roughly $(400 - 700)$nm.

The intensity of light is defined by the number of photons passing through a unit area in a unit of time. That will be the case when using optical microscopes to analyze samples manually by using the eyesight.

Fluorescence occurs as the incoming photons raise the electrons' energy in a fluorophore molecule to an excited state, for instance, in aromatic hydrocarbons. The electron then loses some energy as heat due to the molecule's vibrations, and lastly, the electron returns to the ground state by releasing a new photon with a longer wavelength [26]. This fluorescence process is often illustrated by the Jablonski diagram as shown in Fig. 1.5.
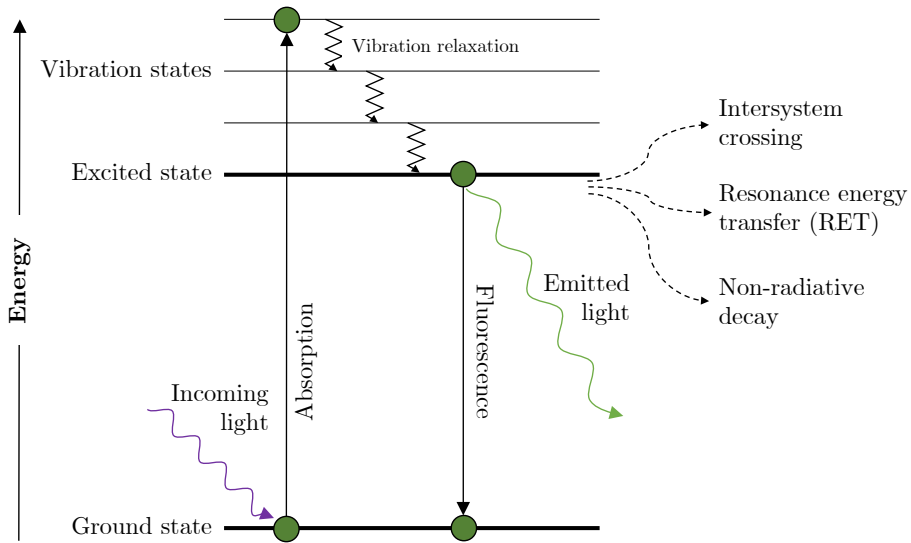
Fig. 1.5: An illustration of a Jablonski diagram, showing the interaction of a fluorophore molecule with a photon with the outcome of an emitted florescent photon.

Since energy has been lost during the process due to vibrational relaxations, the emitted fluorescent photon has lower energy, hence longer wavelength than the excitation photon. Outcomes other than the emission of light can occur, such as non-radiative decay, where energy is released by heat through vibrations, and no photon is emitted. Other less common events are intersystem crossing from the excited state to the triple-state where relaxation from the triplet state to the ground state can occur, also known as phosphorescence [26]. Additionally, resonance energy transfer (RET) is when energy transfers from a fluorescent donor to a fluorescent acceptor when the emission spectrum of the fluorescent donor overlaps with the excitation spectrum of the fluorescent acceptor [27]. Furthermore, the intensity of fluorescence can be decreased by a wide range of molecular interactions, collectively known as quenching. According to Lakowicz [26], quenching can occur by different mechanisms, such as

collisional, excited state reactions, molecular rearrangements, energy transfer, and ground-state complex formation.

The presented fluorescence-based monitor in this thesis detects the content of fluorescent aromatic hydrocarbons in the oil-water-mixture. The fluorescence compound unit is measured as relative fluorescence unit (RFU), which is then transformed to parts per million (ppm) through a calibration curve.
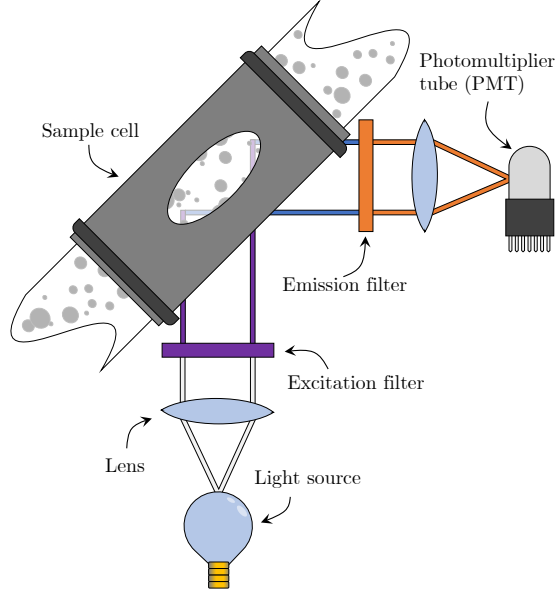
Fig. 1.6: The fluorescence detection structure of the monitor.

Fig. 1.6 illustrates five main components of the fluorescence-based monitor [28]:

- Light source

- Excitation filter

- Sample cell

- Emission filter

- Light detector

Fig. 1.6 shows the working principle of the fluorescence-based monitor for illustration purposes and exact structure and composition of lenses is possibly different [25, 26]. Each component is briefly described in the following paragraphs. Selecting each component depends on the composition of the fluorophore molecule and its fluorescent excitation region; therefore, the components are selected by the manufacturer to detect the type of oil injected into

the experiments.

Light source: The installed near-UV light source emits photons in a wide range of wavelengths. The emitted wavelengths of the light source, published by the Turner Designs [29], is in a range of $\sim(300 - 400)$nm.

Excitation filter: The excitation filter narrows the range of wavelengths emitted by the light source. Although the specific region of the excitation filter is confidential, it is likely in the range of $(300 - 400)$nm according to the document published by Turner Designs [30] and the product numbers of instruments used in this work.

Sample cell: Within the sample cell, the water sample is exposed to the transmitted light after the excitation filter, and the sample ideally emits fluorescence light.

Emission filter: The emitted light passes through the emission filter that narrows the range of wavelengths to ensure only the desired wavelengths are transmitted to the light detector. Again, the emission filter's specific region is confidential, but it is likely in the range of $(410 - 600)$nm.

Light detector: The light detector is a photomultiplier tube (PMT) in the fluorescence-based monitor. The PMT changes the incoming photon into electrons and amplifies the number of electrons. The output signal from the PMT is current pulses. Based on the current pulses, a relation with the concentration of OiW flowing through the sample cell can be established through calibration.

1.1.2 Online Microscopy Analyzers

Continuous online microscopy measurements is a dynamic image analysis method according to International Organization for Standardization (ISO) [31]. This is contrary to the static image analysis method that mostly relates to manual sampling by an optical microscope. Online microscopy utilizes a high-resolution video camera to capture images of the sample stream [2, 16]. These digital images are recorded by a charge-coupled device (CCD) or complementary metal–oxide–semiconductor (CMOS) in the camera converting the readings to a two-dimensional grid of pixels. A photon of light falling on the photodiode of the electronic sensor releases a photoelectron from the silicon photodiode. Pixel-by-pixel, the readout amplifier, converts the photoelectrons into a voltage signal, which is then converted to a digital value. The digital value can then be displayed as a greyscale image on a computer for further analysis [25]. Both online microscopy analyzers used in this work for measuring TSS are similar in their design. An illustration of an online microscopy analyzer is shown in

Fig. 1.7, consisting of a camera, lens, light source, view cell, and a dedicated computer. The computer is installed with a software program that analyzes the images captured by the camera.
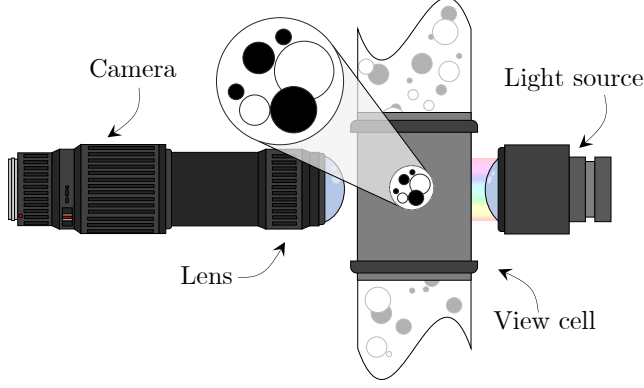
Fig. 1.7: Illustration of a microscopy analyzer's physical parts, consisting of a camera, lens, light source, and a view cell. Figure is the graphical abstract from Paper D.

Both monitors are based on bright field illumination technique, forming a dark image of particles in focus with a bright background. Bacteria can be difficult to capture with a bright field due to being opaque. The specifications of both microscopes are shown in Table 1.1.
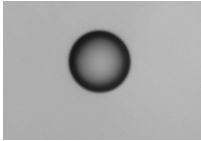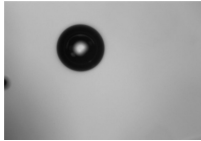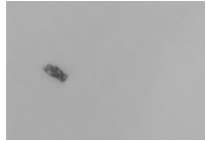
Table 1.1: Specifications of the Canty InFlow and Jorin ViPA. Table is from Paper D.

	Jorin ViPA	Canty InFlow
Pixel length	0.375µm/pixel	0.513µm/pixel
Resolution	1292×964 pixels	1920×1200 pixels
Frame rate	~30fps	~30fps

In order to measure the different properties of the IW, the IW flows through a view cell where images are captured with ~30fps for both online microscopy analyzers. The properties of the TSS can then be measured, such as the particle size, projected area, sphericity, aspect ratio, and equivalent volume. Thus, it is even possible to distinguish between particles and classify them, based on their properties. Table 1.2 shows three different images of captured particles in a process.

Table 1.2: Data information obtained from three different images captured in an experiment.

	Oil droplet	Air bubble	Solid particle
Avg. Feret diameter	72.1616	38.6725	22.9672
Area	4038.1875	1153.9688	291.9375
Perimeter	227.9835	121.8554	75.3347
Aspect ratio	0.9927	0.9975	0.5046
Shape factor	0.9763	0.9766	0.6464
Convexity	1.0102	1.0002	0.9253
Optical density	0.5290	0.7057	0.3195

*Data obtained with Jorin ViPA, based on calibration described in Chapter 4. More property parameters do exist in both microscopy analyzers; this is only a sample of some parameters.

A significant disadvantage of online microscopy analysis is related to the narrow depth of field within the view cell that is captured by the microscopes; thus the measured flow must be a representative homogeneous mixture of the process. An illustration of the narrow depth of field is shown in Fig 1.8.
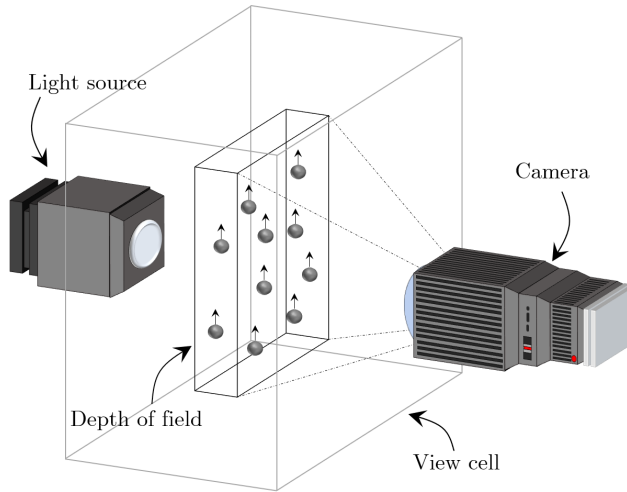
Fig. 1.8: The narrow depth of field within the view cell. Figure is from Paper D.

1.2   Motivation for Online Monitoring

In general, all online quality monitors can presumably be assumed to work well when they are exposed to a known non-hazard environment, and they are properly calibrated (especially by manufacturing personnel) [32–34]. Despite most methods of measuring particles and oil droplets' sizes and concentrations are well-established methods, none has become a standard operation in the oil and gas industry. Although, the Danish Environmental Agency grants every second-year permission of use, discharge, and other disposals of substances and materials, including oil and chemicals in the North Sea, to different ownerships. In the permission, oil and gas companies are obligated to continue logging the OiW concentration by installed online OiW monitors for process optimization [23]. There exist several commercially available monitors for detecting concentrations and sizes of particles and oil droplets, based on different measurement techniques. Some have a broad application focus, where other monitors are designed specifically for the oil and gas industry. Most of the technologies existing today for measuring particle sizes have been developed several decades ago and are commercially distributed by several manufacturers. Therefore, the development of new technologies seems to be scarce, yet the improvement of well-established technologies is still possible. It is also important to realize that the measurement technique is often not the main reason for the error source in the observed results [35]. Merkus [35], Allen [36], and Leschonski [37] all emphasize that the main source of errors are related to sampling and poorly instrument preparation, e.g., improper installment and calibration. The following list highlights multiple issues incountered during the experimental work of this thesis, including calibration and sampling, as mentioned by Merkus, Allen, and Leschonski.

Calibration   The procedure of how the instrument must be calibrated should be thoroughly described. Furthermore, some instruments include too much freedom in the calibration procedure, leading to subjective justification in the calibration procedure. Thus, the instrument's measurement is a direct function of the operator's calibration procedure and will change according to operators.

Sampling   To minimize the uncertainty related to sampling, the procedure must follow the ISO 3171. The sampling procedure is already integrated at most offshore platforms. Nevertheless, the comparison of samples between online monitors and manual reference samples are not documented.

Cleaning and maintenance An automated cleaning procedure is necessary to reduce inconvenient fouling that can reduce the accuracy of the measurement. It is recommended that the cleaning procedure is fully controllable and have the opportunity to sequential execute the cleaning procedure. The maintenance should comply with the manufacturer's request to limit the non-operational period of the instrument. Even worse, if some parts degrade over time, they must frequently be replaced according to the manufacturer's recommendation; consequently, the accuracy of measurement decreases if calibration is not often executed to compensate for the degradation, e.g., the lumen of lamps.

Measurement and reporting errors Measurement errors can occur due to several reasons: particles differentiate from the measurement range, inadequate selection of size distribution model to represent the true distribution, and human error of reporting incorrectly. The latter case's consequence can occur from manual mistakes of labeling correctly or inadequate presentation of the data to an operator. Other measurement errors can also occur due to the instrument's poor electrical installment, vibration, and electrical created noise from other devices or materials.

Instrument's limitations Operators must have sufficient knowledge of the instrument's limitations. This is especially important at the instrument's installment, when calibrating the instrument, and if abrupt changes occur in measurement data.

Performance specifications Knowing the instrument's performance capability concerning the standard operating procedure will strengthen the instrument's reliability.

Reference method To minimize the uncertainty between the reference method and the instrument, it is recommended that they share the same target and even calls for manufacturers to incorporate a manual sampling procedure in relation to the instrument. The performance specification of the reference method (repeatability and reproducibility) in the entire measurement range should be executed for a fair evaluation of the results measured by the instrument and the reference method. Note, the oil and gas industry in

the North Sea follows the OSPAR reference method using a gas chromatography-flame ionization detector (GC-FID) for measuring OiW concentration. There does not exist any regulation for measuring particle sizes in the oil and gas industry, but it is recommended to follow the related ISO standards.

Documentation The user manual and guidance of the instrument, both hardware and software, should be well documented to help the user obtain knowledge about the instrument most sufficiently.

The dominance of the main source of errors listed above is strongly dependent on the type of particles, material properties, concentrations, particle shapes, size variance, and manufacturer. The sum of all errors may often result in doubtful and misleading measurements at field-installations [33, 38, 39]. Measuring the quality of the PW or IW is not only limited to environmental concerns. Seen from an economic perspective, accurate measurements can also benefit from treatment processes, localization of possible contaminates, decision support (i.e., should the PW be discharged or reinjected?), and process optimization, all of which can extend the reservoirs' economic life.

1.3 Paper Motivation

The thesis is divided into two parts: an extended summary and a part containing the papers. The extended summary covers the background and motivation for the project and ties each paper's contributions together. The second part consists of the contributing Papers A−E. An overview of the contributions in terms of papers and how they relate is shown in Fig. 1.9. The remaining of this section describes the motivation for each of the contributing papers.

1.3.1 Motivation for Paper A [2]

The importance of controlling the water quality of the IW is well known in the oil and gas society to be a direct function of injectivity decline and has since the 1940s been known to affect the injection rate [40, 41]. Depending on whether fracturing is undesirable or not, a relatively low concentration of particles can result in rapid reductions in well injectivity. Numerous studies have referenced different plugging tendencies that can affect the injection rate [42–45]. Few of them described that monitoring the process is extremely important to keep a sufficient water quality, e.g., Patton [46], Bennion et al. [47], and Ogden [34], but none of them addresses the complexity of selecting quality monitors that reliably measure the concentration and/or sizes of suspended solids in the

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┌─────────────────────────────────────────┐
│              Paper [A]                   │
│                                          │
│   Online Quality Measurements of Total   │
│     Suspended Solids for Offshore        │
│       Reinjection: A Review Study        │
└─────────────────────────────────────────┘
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┌──────────────────────────────┐   ┌──────────────────────────────┐
│          Paper [B]           │   │          Paper [E]           │
│                              │   │                              │
│  Efficiency investigation of │   │  Human Machine Interface     │
│  an offshore deoiling hydro- │   │  Prototyping and Application │
│  cyclone using real-time     │   │  for Advanced Control of     │
│  fluorescence- and microscopy│   │  Offshore Topside Separation │
│  -based monitors             │   │  Processes                   │
└──────────────────────────────┘   └──────────────────────────────┘
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┌──────────────────────────────┐   ┌──────────────────────────────┐
│          Paper [C]           │   │          Paper [D]           │
│                              │   │                              │
│  Uncertainty Analysis of     │   │  Offshore Online Measurements│
│  Fluorescence-Based          │   │  of Total Suspended Solids   │
│  Oil-In-Water Monitors for   │   │  using Microscopy Analyzers  │
│  Oil and Gas Produced Water  │   │                              │
└──────────────────────────────┘   └──────────────────────────────┘
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Fig. 1.9: Overview of the enclosed papers.

process. To the author's knowledge, Paper A is the first to extensively address how different water quality issues can add to the suspended solid concentration and which types of monitors are good candidates.

1.3.2   Motivation for Paper B [48] and C [32]

As the general trend towards more sustainable production by reducing oil discharge to the ocean, PWRI has gained increasing attention in the last decade. PWRI has the potential to extend the reservoirs' economic life by minimizing water discharge to comply with national regulations. To increase the PWRI ratio, the injected water quality, including both seawater and PW, must be high and consistent. Effective management of retaining a high and consistent quality involves appropriate treatment and monitoring. Monitoring the OiW concentration of PW can also help protect the receiving environment when PW is discharged. To the author's knowledge, no online monitors have been accepted as a reference method for measuring OiW concentration. For the acceptance of online monitors for measuring OiW concentration, accuracy, and robustness must be high. As UV fluorescence monitors are the most widespread application used for online measurement of OiW concentrations in PW offshore, a well-known fluorescence-based monitor in the oil and gas industry was investigated. Paper B focused on the application for continuously calculating the hydrocyclone's efficiency by measuring the OiW concentration. By maintaining a high separation efficiency before discharge or reinjection, two option occurs: increasing the production or maintaining a lower OiW concentration for ac-

commodating PWRI. In joined cooperation, Bram et al. have reinvestigated the hydrocyclone's performance even further in several papers [49–52]. Paper C aimed to evaluate the reliability of measuring OiW concentrations using an online fluorescence-based OiW monitor. Paper C questions if the calibration method was adequate, the uncertainty related to the sampling procedure, as well as a hypothetical question of how reliable the reference method (OSPAR GC-FID) is. Paper C thoroughly evaluates the calibration method on four identical fluorescence-based OiW monitors by statistical analysis of their uncertainties. This heavily contributes to the extent to which the data from the specific monitor can be trusted and which assumptions must be considered if implementing a fluorescence-based OiW monitor.

1.3.3 Motivation for Paper D [53]

Based on the extensive review study in Paper A, online microscopy analyzers were deemed to be the best candidate to measure sizes and concentrations of suspended solids. Two online microscopes familiar in the oil and gas industry were investigated. As the two online microscopes' calibration procedure is highly based on the perception of what image objects are in focus, the procedure for calibrating the three main parameters must be consistent for reducing the calibration uncertainties. The three main calibration parameters that have been evaluated in Paper D are threshold value (THV), edge strength value (ESV)/focus rejection value (FRV), and depth of field. To evaluate both microscopes' performance to measure concentrations in steady-state and real-time, oil was selected as a verification object as the fluorescence-based monitor investigated in Paper C can benchmark both microscopes' ability to measure concentrations. Paper D thoroughly evaluates both microscopes' ability to measure OiW concentrations both in steady-state and real-time. Paper D concluded to what extent online microscopy analyzers can be used for measuring TSS sizes and concentrations accurately, and which reservation must be taken if implementing an online microscopy analyzer for measuring TSS. Furthermore, the estimated particle size distribution (PSD) that is presented to the operator must be a sufficient statistically representation of the true PSD and the uncertainty of the estimated PSD must be kept within a predefined bound. Paper D investigated the minimum number of counted particles to present an adequate PSD within a defined confidence interval and the amount of relative error that is allowed.

1.3.4 Motivation for Paper E [54]

Based on the number of output properties withdrawn from the different online quality monitors, the complexity of displaying the data for an operator comprehensively increases. This will especially be the case for microscopy analyzers as

several properties can be displayed, and each type of particle can be classified. Even if online quality monitors only outputs one value, other additional information could be valuable to be displayed, such as an uncertainty interval from the monitor, if that will help the acceptance of OiW monitors as mentioned in Paper C. Paper E presents a solution to incorporate the graphical design of measured properties most conveniently to an operator, seen from a developer point of view. A connection between Simulink Real-Time and ABB human machine interface (HMI) software via an open platform communications (OPC) server has been established, sending and receiving information. By doing so, new solutions from, e.g., the academic society, can be presented in a familiar environment to industrial partners. That is particularly useful, as it will reduce the concerns related to the solution development in an academic used software, which may not be applicable in a programmable logic controller (PLC) system nor be representable in an HMI software. To demonstrate the established connection's usefulness, a case study was carried out to visualize a developed model predictive control (MPC) solution's behavior. The MPC solution used as a case study is thoroughly described by Hansen et al. [55]; thus, at no given time was the underlying control theory discussed in Paper E.

Chapter 2

# State of the Art of Online TSS Monitoring

In this section, the review article (Paper A) is summarized, covering the main challenges associated with measuring the water quality related to TSS. This section is divided into two parts: Sec. 2.1; injection water characteristics and formation damage mechanisms, and Sec. 2.2; selection of online monitoring for measuring TSS.

2.1 Injection Water Characteristics and Formation Damage Mechanisms

IW has been known for several decades to be a solution to increase the cost recovery ratio [40, 41, 56–60]. The main reason for injecting water into the reservoir is to maintain the reservoir pressure, which naturally drives the production. The second reason is to sweep the reservoir. A common well pattern consists of four injection wells surrounding a production well, forming a five-spot pattern to displace the oil occupied in the pores of the underground porous media toward the production well. If necessary, other patterns can be selected to increase the injection rate, such as a seven-point or nine-point pattern. The IW quality was already observed in the early 1940s to affect the recovery process significantly [2, 40, 41]. Water quality of IW is typically referred to in terms of suspended solids as these directly affects the production of oil from the reservoir [42, 46]. This renders the IW quality problem to be a weighting of net present value, where low operational expenditure (OPEX) must be weighted against longer and more sustainable production. Ideally, the IW should be non-scaling and free of suspended solids and organic matter; thus, the injectivity rate is maintained without any degradation in the reservoirs'

operational lifetime. The IWT process should be protected against corrosion, erosion, and microbiological growth [61]. The pursuit of achieving perfectly clean IW is expensive, to such a degree that operation will seize to be profitable. The geological formation and geographical location of the reservoir are the two factors that determine the physical, chemical, and biological properties of the oilfield [62]. Balancing between the IW quality and the economically profitable estimation of the reservoir lifetime is highly dependent on location and formation. For operators to maintain an adequate IW quality for the specific formation, they must have confidence in the measured data [34, 54, 63]. Even though off-line sampling is often necessary to apply with the reference method, the response time of off-line sampling is long and it can be challenging to maintain a consistent quality during operation. Online installations of proper and reliable monitors will increase the awareness in cases were the process deviates from normal operation behavior within a short period of time compared to off-line samplings [42]. In Paper A, the concerns of measuring the water quality related to TSS were investigated on an IWT facility illustrated in Fig. 2.1.
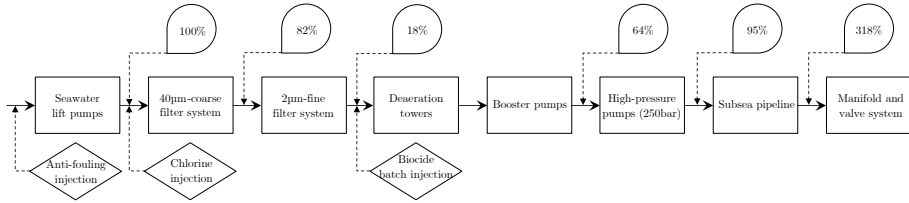

Fig. 2.1: The block diagram illustrating a IWT facility located in the Danish sector of the North Sea. Each percentage represents the TSS concentration measured manually at different locations on the IWT facility. The exact concentrations are confidential. Figure is from Paper A.

As a benchmark, the results that were manually measured from an IWT facility located in the Danish sector of the North Sea raised the importance of investigating IW's characteristics and what type of online monitoring will be most helpful to improve the IW quality. Photos from the manual experiment following Danish Standard (DS) 207 and the results are shown in Fig. 2.2.

With nearly a seventeenfold increment of the TSS concentration mean after the fine filter system to after the subsea transportation pipeline, indicating the addition of TSS such as scales, corrosion deposits, and microbial grows; assuming the filtration systems works as intended. This also concludes that the instrument that measure the particle size distribution should be able to measure within an overall TSS concentration from >30.5mg/L ($\mu + 2\sigma$) to ~0mg/L. As TSS are added within the process, it is difficult to conclude the particles' actual sizes based on the TSS concentration results.

(a)

(b)
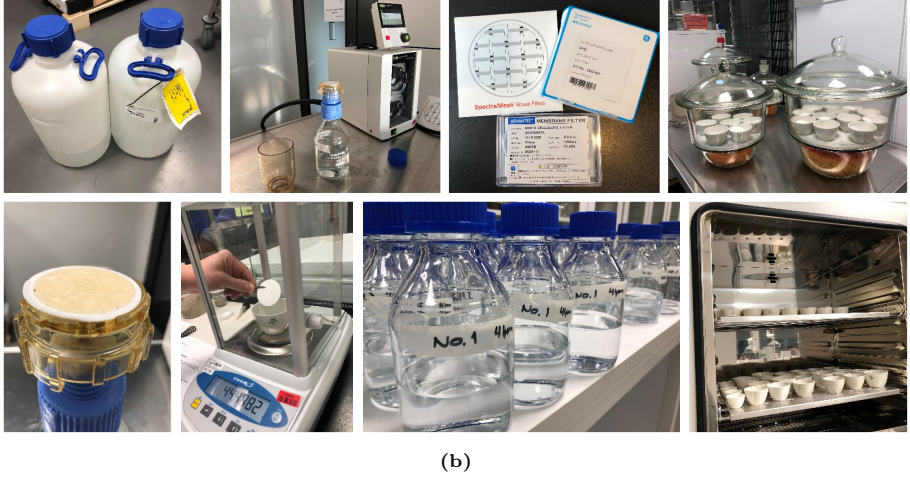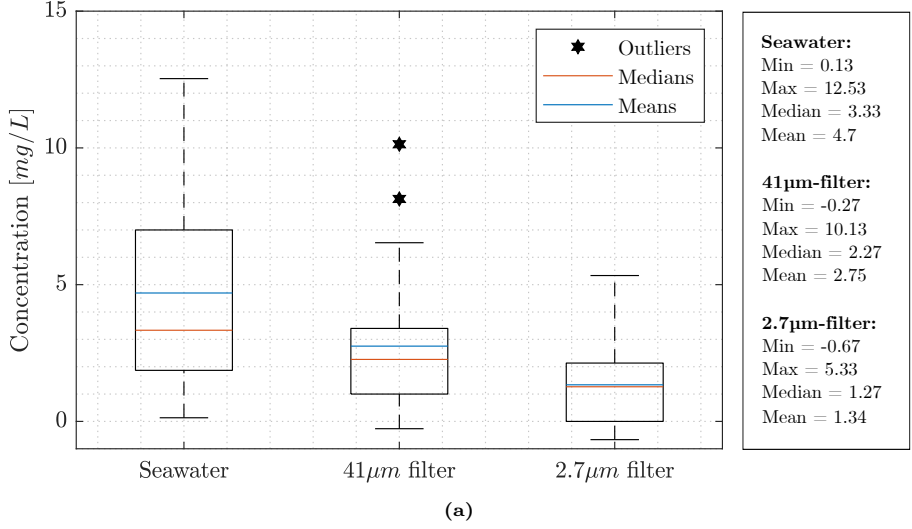
Fig. 2.2: Results and photos of the manual experiment of measuring TSS concentration: (**a**) box plot representation of between each filtration: unfiltered seawater, 41µm-filter, and 2.7µm-filter. Figure is from Paper A; (**b**) photos of the DS 207 experimental execution of the IW from the benchmarked IWT facility.

For determining the different influences of the IW quality, a block diagram is illustrated in Fig. 2.3, followed by a description for each of the IW's characteristics and their formation damage mechanisms.

Other important properties of water are the effects of temperature, acidity, and salinity. The main IW quality characteristics, listed in Fig. 2.3, are summarized in the following list in relation to suspended solids:
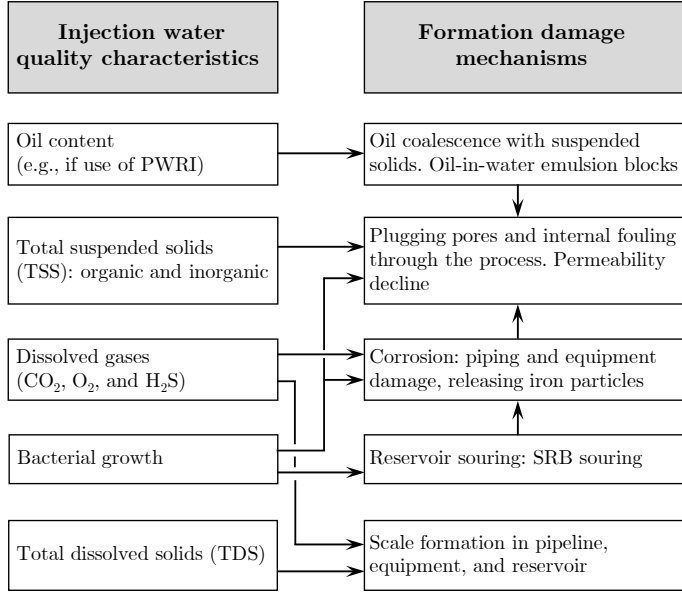
Fig. 2.3: The block diagram illustrates how main IW quality characteristics can negatively effect the injection rate, IWT process, and formation. Figure is from Paper A.

Total suspended solids: The definition of TSS and total dissolved solids (TDS) must be established. There is a general confusion of the definition, as TSS and TDS's definitions are by Baird et al. [64] discriminated by a 2µm filter. The confusion is easily observed in Table 2.1. In the thesis, TSS is defined as anything in the water that can be trapped by a filter, making the definition filter size-dependent [65]. TDS then becomes anything, other than the carrying phase fluid, that passes the selected filter.

Table 2.1: Different definitions of TSS and TDS. Table is from Paper A.

Definitions of TSS and TDS used in different studies	Source
TDS defined as materials that are soluble in water	[16, 34, 66]
TDS defined as materials that passes through a 2µm-filter	[67, 68]
TDS is indirectly defined as materials that are soluble in water	[69–71]
TDS is indirectly defined as materials that passes through a 2µm-filter	[42, 59, 72, 73]
Undefined or too uncertain to tell	[45, 47, 62, 74]

Oil content: Historically, mostly seawater has been used as IW, but due to the increasing environmental awareness, the discharge policies may require PWRI to reduce the discharge of crude oil even further. Especially, as described in Sec. 1.1, the former Mærsk Oil discharged 96% of allowable oil to the ocean in 2017/2018. The increasing attention to PWRI and the presence of oil must be considered when evaluating the IW quality. The presence of crude oil can affect the IWT process and the reservoir in direct and indirect ways such as agglomeration-effect of oil with other matters, asphaltenes deposits, and emulsion blocks [46].

Total dissolved solids: IW with high concentrations of dissolved solids is the foundation of scales in pipelines and instruments. If scales deposit in the process, several issues can occur; it can decrease the injectivity, and more severely completely plug parts of the process. The scales can also deposit into the treatment train, thus, increasing the TSS concentration after the filtration system. Scales often occur when PW and seawater mixes, as seawater can contain significant concentration of sulfate (SO_4^{2-}) and carbonate (CO_3^{2-}), while PW or formation water contains calcium (Ca^{2+}), barium (Ba^{2+}), and strontium (Sr^{2+}) [75].

Dissolved gases: The presence of dissolved gases do not add to the TSS concentration. Similar to TDS, dissolved gases affect the TSS concentration indirectly by promoting an electrochemical reaction between steel and the contacting aqueous phase, creating a corrosive environment that eventually facilitates corrosion deposits that adds to the TSS [34, 76]. The high concentrations of dissolved gases are the main source of corrosion together with growth aerobic organisms that play a role in microbially influenced corrosion (MIC) [77, 78]. The most common dissolved gases that causes corrosion are O_2, CO_2 (sweet), and H_2S (sour) [77, 79, 80]. The presence of O_2 is the most corrosive gas of these three, that is capable of causing serious damage well below concentrations of 1ppm [77, 81–83].

Bacterial growth: When discussing negative influence of microorganisms in IWT processes it is often referred to as MIC, that accelerate the corrosion inside the process, which indirectly adds to the TSS concentration through corrosion deposits. The presence of biofilm and microorganisms above a certain size can also contribute to the TSS concentration directly by physically plugging pores or by bridging. The development of biofilm is shown in Fig. 2.4. Another side-effect of microorganisms in the process is biofouling, which can physically block parts of the process, especially in the filtration system. How much of the corrosion in a process is related to MIC can be challenging to measure, but studies predict that MIC accounts for $(20-30)\%$ in the oil and gas industry, and some studies even predict around 50% [78, 84–86].
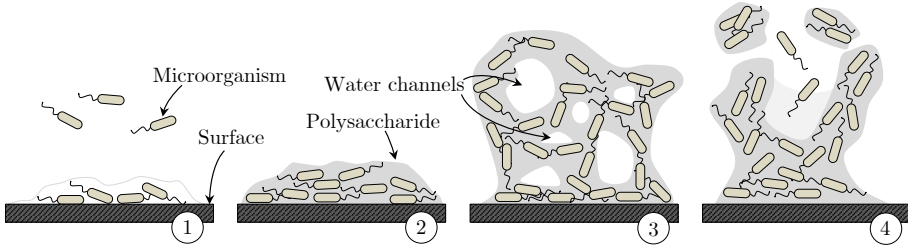
Fig. 2.4: The illustration shows four development stages of biofilm: ① adhering of free floating biofilm producing bacteria to surfaces in the process. ② colonization inside EPS. ③ biofilm is produced and the *in-situ* ecosystem is nourished by water channels to grow [87]. ④ The biofilm reaches critical environmental factors and which the biofilm detaches from the colony. Figure is from Paper A.

2.2 Online Monitoring of Total Suspended Solids

There exist several methods to estimate the concentration and size distribution of both OiW and TSS related to the oil and gas industry. Different measurement techniques have been developed to fulfill the task: ultrasonic spectroscopy, electrical sensing zone, light scattering, light obscuration, and microscopy analysis. Despite the long history of implementing quality monitors for measuring particle sizes, there is no consensus as to which method provides the most reliable estimate, leaving petroleum engineers with a bewildering array of choices [74]. Through a the discrimination process, Paper A concluded that microscopy has the highest potential for measuring particle sizes. The selection was based on two parts:

1. A rough selection based on different measurement techniques, defined by ISO standards, related to measuring particle sizes suspended in a liquid. Table 2.2 highlights if the different measurement techniques apply to on-/in-line installment and their overall size ranges.

2. The second selection was based on the measurement techniques in general pros and cons, without drawing any conclusion on specific manufacturers' equipment.

Two decisive factors favorise microscopy analyzers compared to other online monitors for measuring TSS. As the sample of particles in an IWT process can consist of a wide variety of different matters and diverse sizes, most other methods are challenged by the assumption that even though a size distribution can be obtained fast, the highly sophisticated methods for measuring particles presume perfectly spherical, and the influence of particles' shapes are not taken into account [37]. Many of the other methods are still suitable for measuring particle sizes when the particles of interest are dominant in the process,

Table 2.2: Particle size analysis methods presented in ISO standards. Table is from Paper A.

Particle size analysis' methods	ISO standard(s)	Overall size range [µm]	On-/in-line capable
Sieving	2591-1 3310-1 to -3 20977	$5 - 125$*	✗
Gravitational sedimentation	13317-1 to -4	$0.5 - 100$	✗
Centrifugal sedimentation	13318-1 to -3	$0.1 - 5$	✗
Electrical sensing zone	13319	$0.4 - 1,200$	✓
Laser diffraction	13320	$0.1 - 3,000$	✓
Image analysis methods	13322-1 to -2	$(0.25)1 - 500$*	✓
Small-angle X-ray scattering	17867	$0.001 - 0.14$	✗(✓)
Scanning electron microscopy	19749**	$40.01 - 500$*	✗
Ultrasonic attenuation spectroscopy	20998	$0.01 - 3,000$	✓
Transmission electron microscopy	21363**	$0.001 - 5$*	✗
Light obscuration	21501-3	$1 - 100$	✓
Dynamic light scattering	22412	$0.02 - 3$* ($\ll 10$)	✓

* No typical size range was given within the ISO standards, found according to Merkus [35].

** ISO standard under development.

such as crude oil after the treatment process at the topside production process offshore. Even though microscopy can provide more single-particle properties than most of other methods, microscopy is only more valuable if additional information such as shapes and classification is applied. I.e., an equivalent diameter obtained from microscopy is particularly not more informative than other methods. The second noteworthy advantage of microscopy analysis over other methods is the ability to discriminate captured suspended solids manually. The manual justification is useful for improving the classification procedure and for evaluating the captured images.

Table 2.3 presents a non-exhaustive list of five different microscopy analyzers published in Paper A. Table 2.3 provides a comparative overview of the different microscopy analyzers based on their design and options available. The assortment of microscopy analyzers is based on their diversity in:

- View

- Classification options

- Connection

- Detection range

- Familiarity

The software process performance and ability to acquire samples by each manufacturer cannot be evaluated without any hands-on experience with each equipment, which has not been covered in Paper A. Alternatively, a brief description

Table 2.3: Comparison of five different microscopy analyzers. Table is from Paper A.

Manufacturer		Jorin	J.M. Canty	Grundfos	ParticleTech	SOPAT
Instrument name		ViPA	InFlow	Bacmon	oCelloScope	MM2, Ma
Familiar with the oil and gas industry		✓	✓	✗	✗	(✓)*
Distinguish between solids, droplets, and bubbles		✓	✓	(✓)	(✓)	✓
Categorize different solid types		✓	✓	✓	✓	✓
Distinguish bacteria and abiotic particles		✗	✗	✓	✓	✗
Training classification (neural network, machine learning)		✗	✓	✓	✗	✗
View		2D	2D	3D	3D	2D
Connection		At-line/on-line	At-line/on-line/(in-line)	At-line/on-line	At-line	In-line
Measurement range [µm]		<150**	0.7 − 480	0.6<**	0.5 − 2000	0.5 − 90, 1.5 − 280
Pressure range [bar]		<120	<689	2 − 10	−	0.01 − 3, 0.01 − 320
Temperature range [°C]		<120	−	5 − 40	20 − 40 (operation temp.)	0 − 50
Flow velocity [m/s]		0.03 − 2.1 (0.05 − 4)L/min	0.25 − 2.74	Batch operation, 10min cycle	Batch operation, 10min cycle	−
Frame rate [Hz]		30	30	−	−	15
Cleaning procedure		Manually with flexible stick	Automatically vapor removal system	Flushed between each batch cycle for 1min	Flushed between each batch	Automatically liquid cleaner (Ceramat Sensor Lock-Gate)
ATEX approved		✓	✓	✗	✗	✗, ✓

*Applied at a testing facility related to upstream oil-water separation process. However, to the authors knowledge it has not been installed at a fully integrated upstream separation process.
**Minimum or maximum measurement range is not explicitly defined.

of each microscope's limitations and advantages based on the manufacturer's specifications has been evaluated. By incorporating the advantages from each of the five different microscopy analyzers, such as automatic cleaning procedures, machine learning classification procedures, 3D image analysis, and manually defined particle classes by colors after the analysis, microscopy analyzers can further be improved. Furthermore, a microscopy analyzer system can always benefit from higher resolution, better software design, and more detailed user manuals [2].

Similar to TSS monitors, there are many different techniques and manufacturers for measuring OiW concentrations and droplet sizes; however, a review of those has not been published in Paper A. Several other studies have focused on OiW monitors, with UV fluorescence being the most widespread technique for online measurement of oil in PW [16, 33, 88, 89].

2.3 Conclusion

Water analysis in the oil and gas industry can yield highly informative analytical information to an operator. The analysis can assist in identifying the

quality of production, when contamination appears in parts of the production zone when there is an increasing tendency for corrosion and scale deposition, and for evaluating process design changes. However, for water analysis to be useful, the water analysis must be representatively sampled, and the sample must be withdrawn, analyzed, and interpreted correctly.

This chapter investigated different water quality problems related to TSS that can affect the injectivity rate and the system. As manual water analysis is a time-consuming process, which translates to a slow reaction time, where critical process deviations are observed too late, resulting in process failures. To investigate this, several techniques were evaluated for measuring TSS. Where through a discrimination process based on results in several studies, the author strongly believes measuring different TSS properties with microscopy will bring the most promising results compared to other techniques. Even as several online microscopy analyzers exist on the market, the instrumental design and software design must still further be improved. However, microscopy analyzers' ability to perform shape analysis is the prerequisite for closing the link between the assumption of particles being spherical and the practical results obtained.

Chapter 3

Online Monitoring of Oil-in-Water Concentration

With UV fluorescence as the most widespread application used for measuring the OiW concentration in PW offshore, an investigation of the instrument's reliability was investigated in Paper C. The uncertainty related to the reference method was questioned to have a negative impact on the acceptance of online monitors, moreover the sampling procedure. Four fluorescence-based monitors (Turner TD4100-XDC) was statistically investigated by examining their calibration method, their robustness to different interferences, and their reproducibility between each other. However, the OiW concentrations were not validated according to the reference method (OSPAR GC-FID) in Paper C. Unlike Paper C, Paper B is a typical example of using quality monitors prior to evaluating their performance. Paper B investigates the efficiency of offshore deoiling hydrocyclone in the pilot-plant facility.

The pilot-plant used in both papers is shown in Fig. 3.1, however smaller standalone systems have also been constructed to execute the experiments in Paper C. The pilot-plant has, in the past, been used for other studies to test and develop new control algorithms, validating the separation efficiency of different treatment methods, fault detection and diagnosis, and evaluating the performance of various instruments [52, 55, 90–93].

A piping and instrumentation diagram (P&ID) of the pilot-plant facility is shown in Fig. 3.2, where only the support and hydrocyclone subsystems were used in the experiment executed in Paper B and C.

Fig. 3.1: Photos of the pilot-plant. Left: the pilot-plant; top middle: two of the three online monitor types on a movable skid; bottom middle: the Matlab Simulink Real-Time interface of the pilot-plant; top right: example of the OiW mixture in the buffer tank; bottom right: barrel of the Midland non-detergent Society of Automotive Engineers (SAE) 30 oil used.
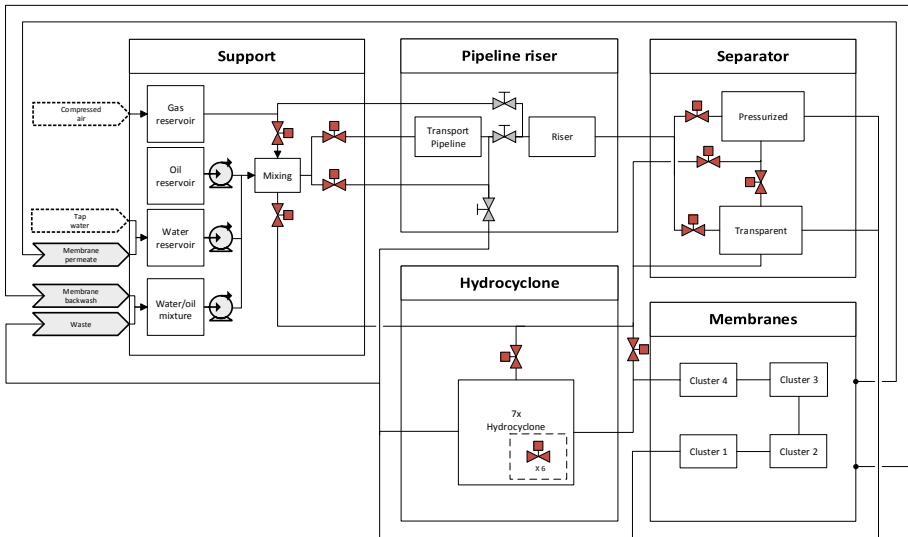
Fig. 3.2: A complete overview of pilot-plant bypass structure at Aalborg University located on Esbjerg campus. Figure is from Paper C.

3.1 Applying Online Oil-in-Water Monitors

With the increasing attention to reducing ocean discharge of oil, obtaining an accurate OiW concentration online is valuable in the pursuit to continuously measure discharged PW. The main priority of online OiW monitors' installment is to fulfill the governmental requirement of oil discharge. Secondly, reliable OiW monitors can be installed at unmanned installations, where currently only one sample set is collected during each planned visit [94]. Another advantage of proper installed OiW monitors is the requirement for new state-of-the-art control solutions and integrated models, published in several studies, that could economically increase the production [52, 92, 95]. However, as discharge regulations are clearly defined, the operation is up to the producers' economic strategy.

On the basis of Paper B, including OiW monitors, it will be beneficial for innovative solutions for increasing the separation process with the use of measured OiW concentration as a feedback signal for control. As hydrocyclones are commonly used as the last separation stage before discharging, it is a natural area to strive for the highest separation efficiency without sacrificing the production rate. Hydrocyclones utilize rotating flow to expose the fluid to large centripetal forces in order to enhance the separation process by forcing the water to the cyclone wall, and the lighter oil will migrate towards the center [49, 96, 97]. The hydrocyclone's separation principle is illustrated in Fig. 3.3.
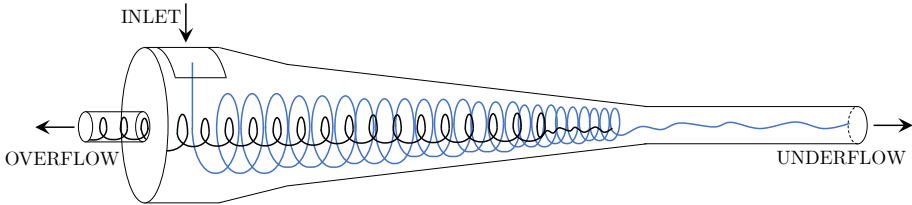
Fig. 3.3: Illustration of an offshore deoiling hydrocyclone consisting of a single liner. Figure is modified from Paper B.

The performance of current offshore hydrocyclones is indirectly controlled by maintaining a pressure different ratios (PDR) [97, 98]. Thus, direct efficiency control for deoiling hydrocyclones will be achievable with OiW monitors' installment before and after the hydrocyclone. Bram et al. [52] have published an extensive joined work on control-oriented modeling for the deoiling hydrocyclone system using OiW monitors for model validation.

3.2 Calibration of Multiple Identical Fluorescence-Based Monitors

Calibration is the process of comparing the measured value from an instrument with a known value. It is highly essential to calibrate the instrument to ensure the measurements have good accuracy. The installation's environment will affect the measuring device; it may be necessary to take precautions if the instrument is highly sensitive to changes, such as harsh environments, vibration, temperature, pressure, and flow. The calibration published in Paper B follows the guideline documented by the manufacturer, as shown in Fig. 3.4, where at least two samples must be taken [28].
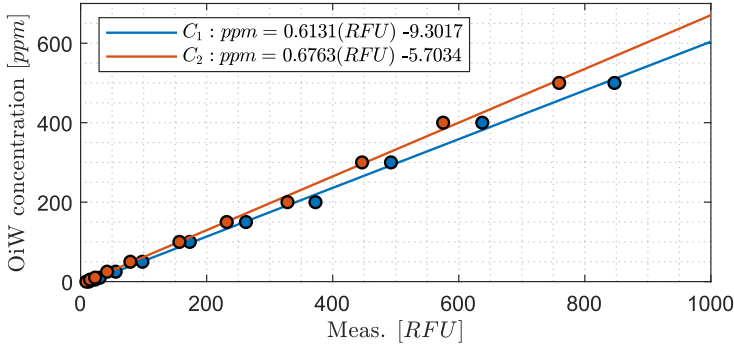
Fig. 3.4: Ordinary least square calibration regression based on eleven measured RFU values for both fluorescence-based monitors. Figure is modified from Paper B.

Multiple points are necessary to verify whether the relation follows a linear or nonlinear trend. For both calibration in Paper B and C, the relationship between measured RFU and injected OiW concentration follows a linear trend. The calibration procedure in Paper B was executed on two identical fluorescence-based monitors within a calibration range of $(0 - 500)$ppm, and in Paper C the calibration range is $(0 - 300)$ppm, as shown in Fig. 3.5. However, below 10% of the entire RFU range was only covered, which may have challenged the instrument's sensitivity in Paper B. For Paper C, 400ppm was chosen as the highest value of interest by adjusting the instrument's sensitivity. 400ppm were selected as the OiW concentration before entering the deoiling hydrocylone based on different studies [99, 100].

In Paper B, the default calibration procedure documented by the manufacturer uses ordinary least square (OLS) regression to fit the measured RFU value with the injected OiW concentration; thus, a homoscedastic random error must be assumed. Paper C investigated whether OLS regression is the most optimal calibration procedure. The results using OLS and weighted least

square (WLS) was compared in Fig. 3.5, based on the same 70 sample points for each of the four identical monitors at the following OiW concentrations: $(0, 10, 20, 50, 100, 150, 300)$ppm, ten samples for each concentration.
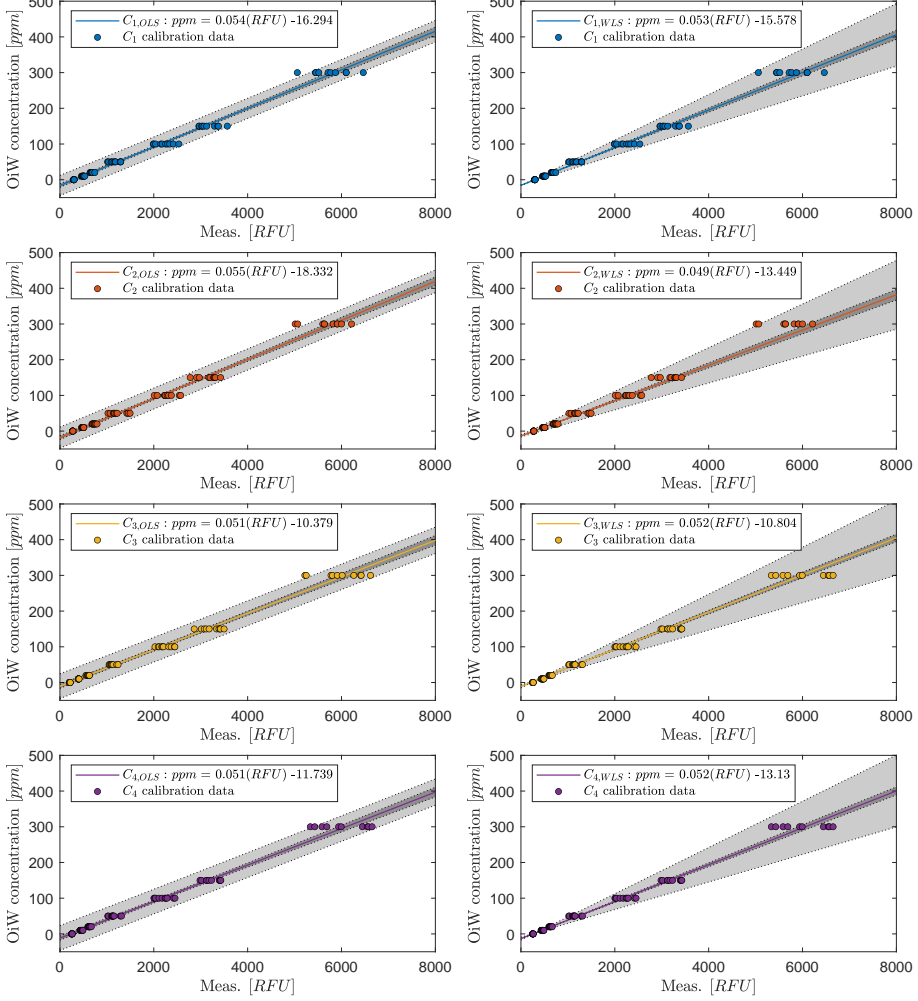
Fig. 3.5: Two different least square regression procedures for each of the four OiW monitors. Left column: linear regressions using OLS; right column: linear regressions using WLS. Figure is from Paper B.

Due to fairness, the procedure presented by the manufacturer and shown in Fig. 3.4 was not executed for OLS. Even though the weighting factor (w_i) for $i = 0, 1, 2..., n$ does not affect the regression result using OLS, including the number of samples (n) will still positively affect the calibration procedure. The

most significant disadvantage of WLS is determining the w_i, as w_i is unknown. Using the reciprocal of the variance (σ^{-2}) for each calibration concentration is a reasonable candidate of w_i.

Fig. 3.5 is mathematically presented in equations: $(3.1)-(3.13)$. A more detailed description of the equations can be found in Paper C, where

$$y = ax + b, \tag{3.1}$$

$$b = \frac{SS_{xy}}{SS_{xx}}, \tag{3.2}$$

and

$$a = \overline{y} - b\overline{x}. \tag{3.3}$$

a and b are the slope and the intercept on the ordinate axis, respectively. SS_{xx} and SS_{xy} are the weighted sum of squares and the weighted sum of cross-products, respectively, and $\overline{x}$ and $\overline{y}$ are the arithmetic weighted means. To calculate the prediction interval (PI) and confidence interval (CI), the following equations are used:

$$\overline{x} = \frac{\sum_{i=1}^{n} w_i x_i}{\sum_{i=1}^{n} w_i}, \tag{3.4}$$

$$\overline{y} = \frac{\sum_{i=1}^{n} w_i y_i}{\sum_{i=1}^{n} w_i}, \tag{3.5}$$

$$SS_{xx} = \sum_{i=1}^{n} w_i (x_i - \overline{x})^2, \tag{3.6}$$

$$SS_{xy} = \sum_{i=1}^{n} w_i (x_i - \overline{x})(y_i - \overline{y}), \tag{3.7}$$

$$SSE = \sum_{i=1}^{n} w_i (y_i - \hat{y})^2, \tag{3.8}$$

$$\hat{s} = \sqrt{\frac{SSE}{DOF}}, \tag{3.9}$$

$$CI = ax_0 + b \pm t_{0.025} \hat{s} \sqrt{\frac{1}{n} + \frac{(x_0 - \overline{x})^2}{SS_{xx}}}, \tag{3.10}$$

and

$$PI = ax_0 + b \pm t_{0.025} \hat{s} \sqrt{\frac{1}{w_0} + \frac{1}{n} + \frac{(x_0 - \overline{x})^2}{SS_{xx}}}. \tag{3.11}$$

SSE is the sum of squared errors, $\hat{s}$ is the predicted sample standard deviation, DOF is the degrees of freedom, and $t_{0.025}$ is the t-score with two-tailed 95%

confidence. The only difference between OLS and WLS is w_i. For OLS and WLS, $w_i = 1$ and $w_i = s_i^{-2}$, respectively. The notation "$\wedge$" denotes predicted value and the subscript "$_0$" denotes the set of observations. As the true variance of the calibration region is unknown, w_0 is estimated based on the predicted variance function:

$$s(\hat{y}) = c_1 \hat{y} + c_0 \tag{3.12}$$

and

$$w_0 = s^{-2}(\hat{y}), \tag{3.13}$$

where c_0 and c_1 are obtained from least square method on known sample standard deviation (s_i) in $y_i = (0, 10, 20, 50, 100, 150, 300)$ppm. Eq. (3.12) may only be a suboptimal representation of the variance function for the OiW monitor, alternative candidates to represent the variance function are published by Noblitt et al. [101]. Whether WLS is a better calibration procedure than OLS is evaluated based on the experimental results in Fig. 3.6. The evaluation is executed on the same 100min experiment with addition of oil in steps starting at 0ppm: (5, 10, 20, 40, 80, 160, 320, 400)ppm.

The deviation between the OiW monitors calibrated using WLS was in every OiW concentration smaller compared to calibration with OLS, as shown in Table 3.1 and observed in Fig. 3.6. This is especially evident at low OiW concentrations where the deviation of WLS is two to three times smaller than with OLS. Whereas, at higher OiW concentrations, the deviation between both methods is low. This tendency agrees with the theory that a linear unweighted regression tends to fit points at the upper calibration levels compared to lower calibration levels due to the square errors measured in absolute values.

Table 3.1: Data results from the experiment shown in Fig. 3.6. Table is from Paper C.

X_{pred} [ppm]	Biggest div. between $\overline{C}_1, \overline{C}_2, \overline{C}_3, \overline{C}_4$ with OLS [ppm]	Biggest div. between $\overline{C}_1, \overline{C}_2, \overline{C}_3, \overline{C}_4$ with WLS [ppm]	Biggest div. from $\overline{\overline{C}}_{OLS}$ [ppm]	Biggest div. from $\overline{\overline{C}}_{OLS}$ [%]	Biggest div. from $\overline{\overline{C}}_{WLS}$ [ppm]	Biggest div. from $\overline{\overline{C}}_{WLS}$ [%]
0	4.34	1.56	2.30	–	0.86	–
5	3.43	1.54	1.95	35.1	0.82	13.8
10	2.55	1.70	1.64	15.8	1.00	9.4
20	2.26	1.54	1.50	8.0	0.82	4.4
40	3.31	2.10	1.86	5.0	1.08	3.0
80	7.55	5.33	4.76	6.5	2.85	4.0
160	17.42	13.46	11.21	7.5	7.64	5.3
320	36.41	33.88	24.75	7.8	21.42	6.9
400	55.14	52.91	34.46	8.3	33.83	8.4
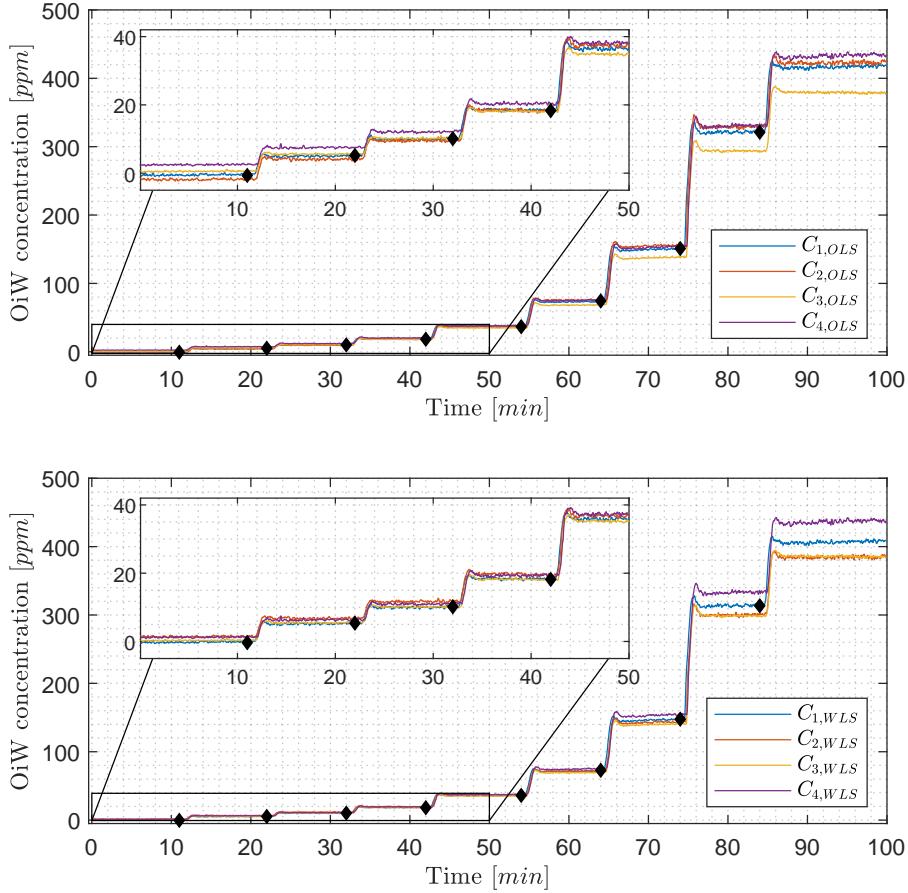
Fig. 3.6: Eight times oil was added with the intention to achieve the OiW concentrations: $(5, 10, 20, 40, 80, 160, 320, 400)$ppm starting at 0ppm, during the 100min experiment. The black diamonds mark when each different OiW concentrations were added. The top located plot shows the results using OLS as calibration procedure, and the bottom located plot shows the results using WLS as calibration procedure. Figure is modified from Paper C.

3.3   Uncertainties

Uncertainties will always be present in measurement results; however, the uncertainty sources' influences vary greatly depending on the procedure, instrument, and environment. The magnitudes of the deviations caused by uncertainty sources are usually unknown and can thus only be estimated. In this section, different uncertainties related to measuring OiW concentration with fluorescence-based monitors are addressed. The combined uncertainty using

the GC-FID reference method, with a 95% confidence interval, can be calculated based on data presented in ISO 9377-2 [102], without introducing the uncertainty related to sampling from the production target. Based on the data, between $127 - 156$ number of results were obtained in $35 - 41$ laboratories on four different OiW concentrations ranging between $(0.57 - 3.61)$mg/L. If assuming the oil producers use the same laboratory and personal, and the uncertainties of the reference method are scalable in the entire range of interest, then the uncertainty by the reference method lies within:

$$OiW_{repeat.} = x \pm (5.9 - 27.6)\%, k = 1.96, norm, \qquad (3.14)$$

where k is the coverage factor assuming the uncertainty is normally distributed. The uncertainty increase if different laboratories and personal are used to measure the OiW concentration obtained:

$$OiW_{reprod.} = x \pm (18.8 - 79.4)\%, k = 1.96, norm \qquad (3.15)$$

The data from ISO 9377-2 and the calculation of the uncertainties are described in Paper C. Even though the reproducibility value may not be constant in the entire range of interest, the reproducibility value certainly includes noteworthy uncertainties that should be addressed to promote the industrial use of online OiW monitors by quantifying the trustworthiness of the measurements.

3.3.1 Sampling

By recapitulating the reference method's previous conditions, the uncertainties related to extracting a representative sample, transportation, and storage of heterogeneous oily PW were not addressed. Uncertainties related to sampling are often dominant and occasionally may exceed 90% of the total measurement uncertainty [103]. Another study by Lava et al. [104] states that the sampling uncertainty may influence 75% of the total variance of measurement uncertainty [104]. However, estimating the uncertainty related to sampling can be challenging, but the uncertainty associated with the sampling process must inevitably contribute to the total uncertainty of the measured OiW concentration [103]. A great deal of minimizing of sampling uncertainty has been done by standardizing the sampling procedure in ISO 3171 [105], whereas storage and transportation of the sampling should follow the ISO 9377-2 [102] and ISO 5667-3 [106]. To give a certain understanding of where different uncertainty sources can occur from in the standards for PW, a list of different procedures that must be fulfilled before an analysis can can be executed onshore:

- The sample should be taken from rising flow in a vertical pipe section, with an isokinetic center lined pitot, in a fully developed turbulent region.

- The distance between the intake opening of the sampling pitot tube and the sampling end-point to where the sample is bottled should be as short as possible to avoid dead volume in the sampling system.

- PW must be flushed through the sampling end-point at least three times to ensure potential residing residue replacement from prior measurements.

- The sample should be preserved by acidifying it to a low pH (pH<2) to kill bacteria that can degrade the oil if the sample cannot be analyzed immediately.

- The sample must be bottled and stored in the absence of light and cooled to $(4 - 8)$°C, to avoid the growth of bacteria, but also to directly avoid the possibility of degradation of petroleum components by photochemical reaction [106, 107].

If the last two bullet points about storage and transportation are disregarded, the uncertainty related to sampling between the manual GC-FID reference method and an online OiW monitor can be reduced greatly if the same sample is measured by sharing target at the exact same time.

3.3.2   Influencing Properties Related to the Fluorescence-Based Monitors

Two different influencing properties were investigated in Paper C: the presence of gas bubbles and flow rate changes. Other known influencing properties from the literature that can affect fluorescence-based monitors are:

- The ratio between aromatic and aliphatic hydrocarbons.

- Inner filter effects.

- Chemical quenchers.

- Degradation of physical parts over time.

Some of the influences have been discussed in Paper C.

Gas bubbles' influence on an OiW monitor: The presence of air bubbles is known from several studies to influence the measurement results of many quality monitors [33, 108–112]. The same was suspected of the fluorescence-based monitors in Paper C, as a false high measurement was observed when visible air bubbles were apparent in the buffer tank in the start-up of each experiment and slowly converged as air bubbles slowly dissipated to the surrounding environment. According to literature, the change in fluorescence intensity with respect to the presence of air bubbles occurs as the fluorescence spectrum is distorted by the scattered light caused by gas bubbles [112].
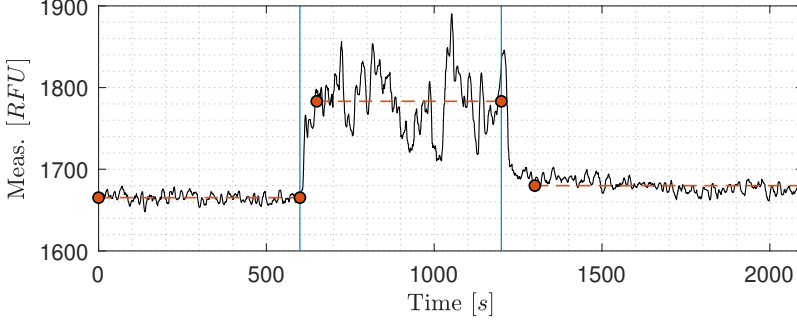
Fig. 3.7: The results measured by the OiW monitor. Gas bubbles are injected in the time period $(10-20)$min of the 35min experiment. Figure is from Paper C.

Fig. 3.7 shows the results of introducing air bubbles into the process. The experiment's execution consisted only of tap water to minimize the number of unknown influences, such as a shift in the volume of oil droplets entering the OiW monitor. Air was consistently added into the process from 600s to 1200s as shown in Fig. 3.7. In the period of injecting air into the system, the RFU measurement increased by 118RFU equivalent to 6.2ppm if using the WLS calibration procedure presented in Fig. 3.5. It is noteworthy to highlight that when only air was present in the view cell (with some uncertainty to small micro/macro water droplets), the measurement was $(80-90)$RFU. This insinuates a sweet spot when the presence of air bubbles by volume overtakes its effect of distorting the fluorescence spectrum. As only tap-water was used in the experiment, further investigation should examine different OiW concentrations with the presence of gas bubbles and how the volume and sizes of gas bubbles influence the results.

Repeatability investigation of flow-dependency on an OiW monitor: As previous joined studies by Bram et al. [52] suspected the OiW monitors to be flow-dependent, an investigation was carried out in Paper C. One OiW monitor was gravity fed with both tap water and demineralized water at two different flow rates: 1.1L/min and 1.7L/min, as shown in Fig. 3.8. Both flow rates were selected as they lie within the recommended range of $(1-2)$L/min by the manufacturer. Each box in Fig. 3.8 shows the median, 25th and 75th percentiles. The whiskers at each box represent the most extreme data points that are not considered as outliers.
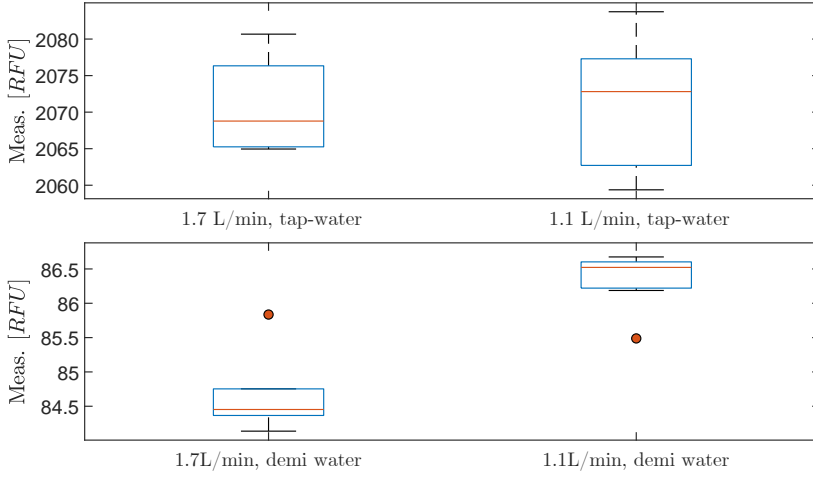
Fig. 3.8: The results of gravity feeding an OiW monitor with both demineralized water and tap water at two different flow rates: 1.1L/min and 1.7L/min. Figure is from Paper C.

The results showed insignificant flow-dependency at both types of water, with a difference in grand median of 4.0RFU for demineralized water and of 2.1RFU for tap water. Equivalently, if WLS calibration is considered, the difference in grand medians in ppm values for both types of water will be 0.2ppm and 0.1ppm, respectively. Although the influence of the RFU readings is insignificant compared to, e.g., air bubbles, the experiment interestingly showed the opposite, as was documented by Bram et al. [52], that the measurement of RFU is lower with a high flow rate. As concluded in Paper C, the suspected flow-dependency discussed by Bram et al. [52] must occur due to insufficient representation of the process flow rather than a change in flow rate.

3.4   Conclusion

An evaluation of four identical fluorescence-based monitors (Turner TD4100-XDC) was executed to determine the precision and sensitivity to measure aromatic oil content in water. The calibration method of the OiW monitor revealed that WLS yields a higher reproducibility between the four monitors, compared to the OLS method. Additionally, uncertainties related to sampling, calibration, and some variation in the process parameters were investigated. The latter uncertainties concerns: flow-dependency and the presence of gas bubbles in the flow stream. The results showed that the fluorescence-based monitors are not or at least insignificantly flow-dependent within its recommended flow rate range by the manufacturer. The results of gas bubbles showed a notably increase of the OiW concentration by 6.2ppm. However, further investigation

must be examined to prove the relation between gas bubbles and OiW concentrations. Whether the OiW monitor is feasible to measure OiW concentrations accurately in highly dynamic separation facilities with continuous fluid composition changes is questionable. Nevertheless, they are still of interest for measuring the separation efficiency of the hydrocyclones to enhance their deoiling performance, assuming the parameter effects on the OiW monitors are scalable.

Chapter 3.   Online Monitoring of Oil-in-Water Concentration

Chapter 4

Online Monitoring of Total Suspended Solids

Based on the conclusion in Chapter 2, microscopy is believed to be the best candidate for measuring TSS in an IWT facility due to their direct observation that can measure single-particle properties and thereby able to classify particles [37]. The specifications of the two online microscopy analyzers: Jorin ViPA and Canty InFlow, are shown in Table 1.1. The calibration method of both microscopy analyzers was examined in Paper D to highlight the subjective assumptions related to calibrating the equipment. Paper D proposed one solution of how the calibration procedure could be performed to increase reproducibility related to the calibration uncertainties. The fluorescence-based monitor, described in Chapter 3, was used as a benchmark to evaluate both online microscopes' performance to measure different OiW concentrations accurately and in real-time.

The test setup used for evaluating both microscopes is shown in Fig. 4.1. The setup is equipped with a centrifugal pump, flowmeters, pressure transmitters, and control valves. The setup is designed for the purpose of testing quality monitors that is installed on-line (sidestream). One significant advantage of online sampling is the applicability regardless of process dimensions in size and flow rate, however, a large number of images is required to yield representative sample of the mainstream [2]. In Paper D, all three quality monitors (Jorin ViPA, Canty Inflow, and Turner TD-4100XDC) were equipped in series on a sidestream. By manipulating the control valves, the setup is configurable to direct the liquid through the sidestream with a constant flow rate within the recommended flow velocity of each quality monitor.

Fig. 4.1: Photos of the skid-mounted test setup. Left: the test setup for testing different online quality monitors; top middle: electrical control panel; bottom middle: the supply tank with a mounted mixer; right: calibration particles manufactured by BS-Partikel.

4.1 Calibrating Online Microscopy Analyzers

For calibrating and validating both online microscopes, known polystyrene particle sizes produced by BS-Partikel were used. A known particle size with a μ and σ of 20.1(0.4)µm was selected for calibrating both microscopes. Two other known particle sizes were used for validating the calibration: 9.8(0.3)µm and 40.3(0.9)µm.

To calibrate both microscopes, three calibration parameters must be tuned: THV, ESV/FRV, and depth of field. Although, tuning the virtual depth of field is only necessary when concentration measurements are needed. The THV consists of values in the range of $0-255$, from black to white. The ESV/FRV are edge detection methods to determine the particles' edge. Jorin ViPA's ESV is in a range of $0-10$, where Canty InFlow's FRV ranges from $0-1000$. The virtual depth of field determines the volume of captured images to match the known concentration.

Selecting THV and ESV/FRV is based on the petroleum engineers' perception of which objects are considered adequately in focus when calibrating the microscope. Fig. 4.2, presents eleven different particles with the same nominal size captured by Jorin ViPA. The numbers in Fig. 4.2 represent the range of ESV. Fig. 4.2 shows particles in different degrees of focus, from where the subjective assumption must be made; which particles are considered sufficiently sharp and at which greyscale value of the particles' periphery should be included to represent its area.

One solution for calibrating the microscopy analyzers was suggested in Paper D, to keep a temporary high ESV followed by adjusting the THV until

Fig. 4.2: Eleven different particles captured by Jorin ViPA each with the corresponding ESV ranging from $0 - 10$. Figure is from Paper D.

the PSD matches the distribution of the known particle size. When increasing ESV/FRV less particles will be captured, and when decreasing ESV/FRV more particles that are less in focus can be underestimated. This complicates the selection of ESV/FRV as these tradeoffs are chosen by the petroleum engineer. However, as the measurement of particle sizes is dependent on the THV and ESV/FRV, other values may yield a better representation of the PSD. The calibration results of the known particle size: 20.1(0.4)µm, from each microscope is shown in Fig. 4.3 and 4.4.

The calibration method was validated by recirculating two other known particle sizes together with the known calibration particle size: 9.8(0.3)µm, 20.1(0.4)µm, and 40.3(0.9)µm. The μ and s for each known particle size, measured by Jorin ViPA and Canty InFlow are shown in Table 4.1. The data were obtained by truncating the entire experiment results at each known particle size with a duration of ±4µm from the known μ by the manufacturer.
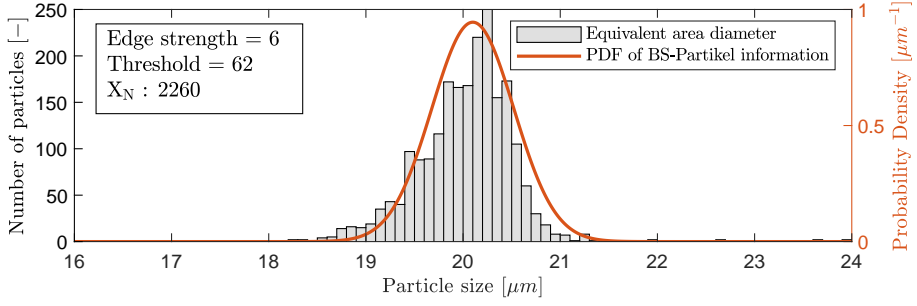
Fig. 4.3: The results of the selected calibration parameters: THV = 62 and ESV = 6 for Jorin ViPA. Figure is from Paper D.
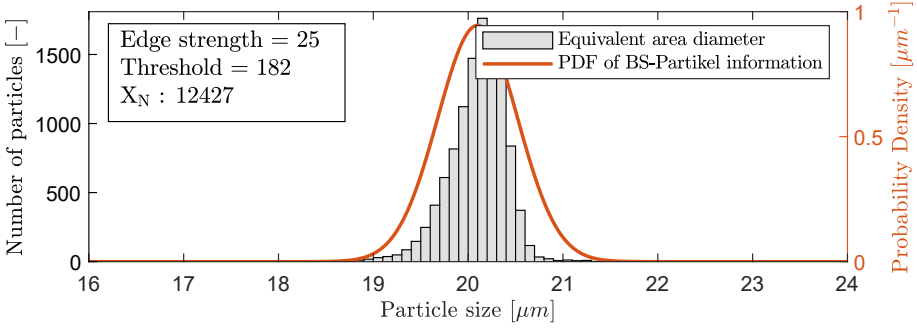
Fig. 4.4: The results of the selected calibration parameters: THV = 182 and ESV = 25 for Canty InFlow. Figure is from Paper D.

Table 4.1: The μ and σ results of recirculating three known particle sizes for 2h. Table is modified from Paper D.

	Known $\mu(\sigma)$ [µm]	$\mu_{estimat.}$ [µm]	$\sigma_{estimat.}$ [µm]
	9.8(0.3)	9.5	0.6
Canty InFLow	20.1(0.4)	20.0	0.6
	40.3(0.9)	40.9	0.5
	9.8(0.3)	9.6	0.6
Jorin ViPA	20.1(0.4)	20.3	1.0
	40.3(0.9)	41.4	0.6

4.2 Performance Validation

Both microscopes' ability to measure concentrations based on classified particles was evaluated. Oil droplets were selected as verification object as the

fluorescence-based monitor was available to benchmark their ability to measure the OiW concentration. A 6h experiment was executed on the test setup as shown in Fig. 4.1. To ensure consistency in the PSD throughout the experiment, the pump speed was fixed to maximum speed, and the flow rate was maintained constant through the sidestream by manipulating the control valve after the quality monitors. Six nominal OiW concentrations were analyzed $(55, 100, 150, 200, 250, 400)$ppm, by injecting oil between each concentration. Fig. 4.5 shows the results of measuring the OiW concentration in steady-state, by outputting the mean concentration every minute. The results in Fig. 4.5 are shown in Table 4.2.
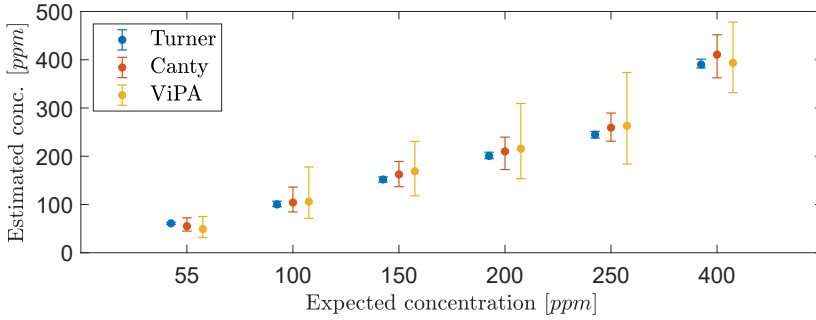
Fig. 4.5: Each error bar represents the steady-state measurement obtained with both microscopes each minute. The error bars shows the μ, minimum, and maximum OiW concentration of a 30min duration for each OiW concentration. The error bars of the fluorescence-based monitor are based on measurements every 10s. Figure is from Paper D.

Table 4.2: Associated results to Fig. 4.5. Table is modified from Paper D.

Expected conc.		55ppm	100ppm	150ppm	200ppm	250ppm	400ppm
	min:	56.2	94.7	146.1	193.9	237.6	379.0
Turner TD-4100XDC	μ:	60.9	100.1	151.8	200.7	244.6	390.0
	max:	67.7	105.4	159.5	207.7	255.8	398.4
	min:	31.6	71.4	118.0	153.5	183.7	331.9
Jorin ViPA	μ:	49.2	106.1	168.9	215.7	263.1	393.6
	max:	75.1	177.8	230.7	309.3	373.4	478.0
	min:	44.9	84.6	136.9	172.4	231.2	362.6
Canty InFlow	μ:	55.1	104.0	162.4	210.0	259.3	410.6
	max:	72.3	136.1	189.2	239.6	289.4	451.7

Both microscopes showed promising results in measuring OiW concentrations in real-time. Fig. 4.6 and 4.7 show the real-time measurement at the expected 55ppm OiW concentration. A trailing moving average window of 1min

was used, although extending the averaging window will naturally smooth the measurements. However, depending on the plant's dynamics, important information might be lost by selecting a too large window.
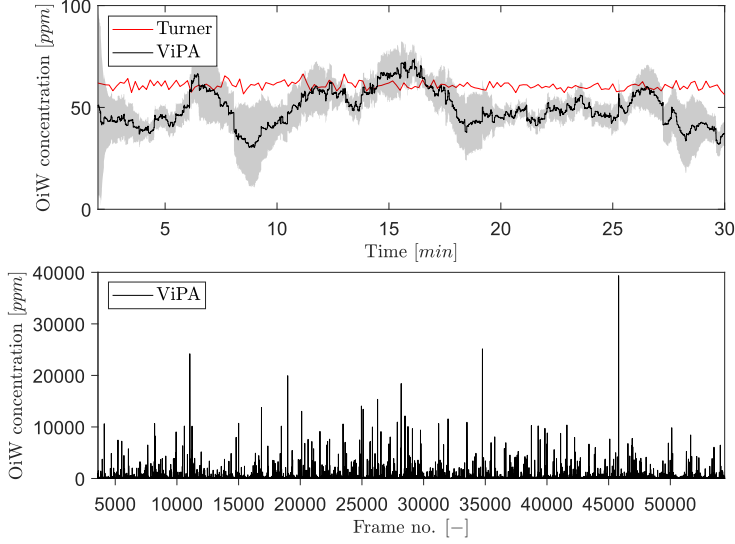
Fig. 4.6: Real-time OiW concentration measurement estimated by Jorin ViPA at the expected OiW concentration of 55ppm. A 95% confidence interval of the averaging window is shadowed behind the signal. The bottom graph shows the volume based OiW concentration in each image captured. Figure is from Paper D.

As the number of captured particles affects the accuracy of the PSD it is necessary to sample a sufficient amount of particles. The estimation of the number of particles that must be counted to achieve a given statistically accuracy was investigated in Paper D. As an example Fig. 4.8 and 4.9 represent the PSD measured by the two microscopes at an expected OiW concentration of 55ppm. Furthermore, the mean and standard deviation of the log-normal distributions, μ_0 and σ_0, is presented in Fig. 4.8 and 4.9, together with the number of counted particles, X_N.

Masuda and Iinoya [113] proposed a mathematical procedure to determine the minimum number of particles that must be counted, n^*, within a defined confidence interval, u, and the amount of error, δ, allowed. Thus, the relative error to a certain time can be estimated based on number of particles observed. n^* is determined by:

$$\log(n_*) = -2\log(\delta) + \log(\omega), \tag{4.1}$$

where

$$\omega = u^2\alpha^2 s^2(2c^2 s^2 + 1). \tag{4.2}$$
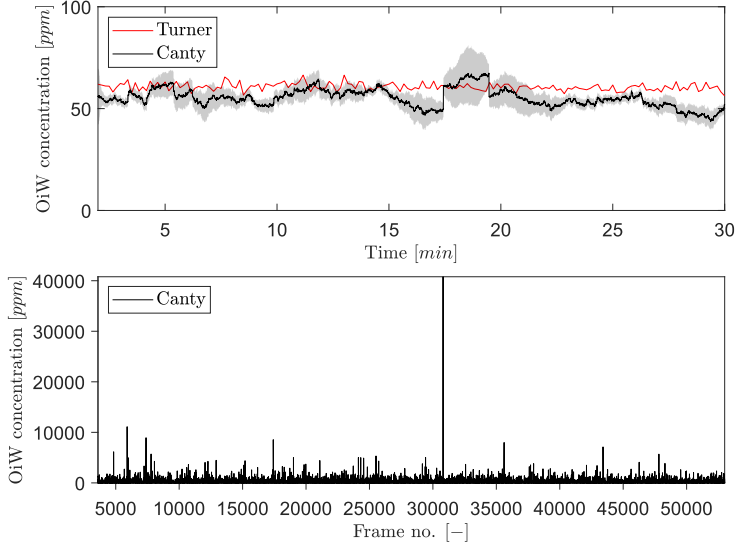
Fig. 4.7: Real-time OiW concentration measurement estimated by Canty Inflow at the expected OiW concentration of 55ppm. A 95% confidence interval of the averaging window is shadowed behind the signal. The bottom graph shows the volume based OiW concentration in each image captured. Figure is from Paper D.
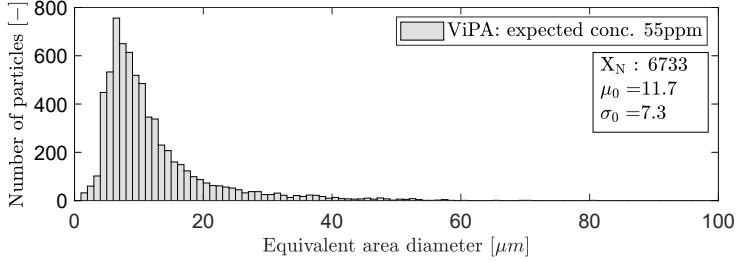
Fig. 4.8: The PSD obtained by Jorin ViPA based on the expected concentration of 55ppm. Figure is from Paper D.

α is a non-zero exponential constant (for log-normal distribution $\alpha = 2$) [114]. Lastly, the constant, c, is:

$$c = \beta + \frac{\alpha}{2} \qquad (4.3)$$

where β is a basis number, for the count basis $\beta = 0$ [53]. Table 4.3 shows the relative error within 95% confidence interval of the PSDs for each measured OiW concentration, based on different obtained time intervals.
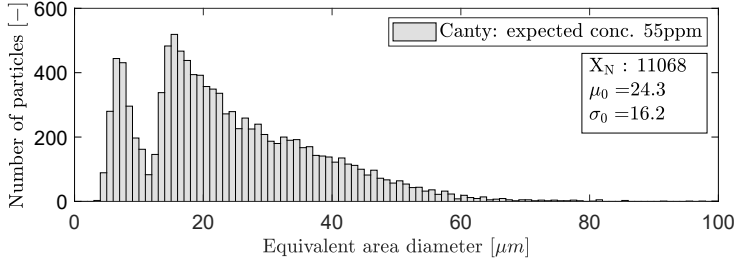
Fig. 4.9: The PSD obtained by Canty InFlow based on the expected concentration of 55ppm. Figure is from Paper D.

Table 4.3: Measured δ within 95% confidence interval based on different durations for each OiW concentration measurement of both microscopes.

Expected conc.		55ppm	100ppm	150ppm	200ppm	250ppm	400ppm
	1min:	19.8%	16.9%	13.7%	13.2%	12.2%	10.5%
Jorin ViPA	5min:	8.6%	7.7%	6.3%	5.7%	5.2%	4.5%
	10min:	6.1%	5.3%	4.5%	4.0%	3.7%	3.3%
	30min:	3.5%	3.0%	2.6%	2.3%	2.2%	1.9%
	1min:	15.4%	13.6%	11.0%	9.2%	1.7%	1.5%
Canty InFlow	5min:	7.5%	6.4%	5.5%	4.8%	4.5%	4.1%
	10min:	5.3%	4.5%	3.8%	3.3%	3.1%	2.9%
	30min:	2.98%	2.6%	2.2%	1.9%	1.8%	1.6%

The results in Table 4.3 shows the potential δ of presenting the PSD within a 95% confidence interval, based on the number of particles captured by both microscopes. If $\delta < 5\%$ is sufficient, the time interval of presenting the data must be at least 5min, although at lower concentrations the duration must be further increased. For obtaining a PSD with $\delta = 1\%$, approximately $100,000 - 130,000$ particles for Canty InFlow and $90,000 - 100,000$ particles for Jorin ViPA must be counted based on the measured OiW concentrations.

4.3   Conclusion

Both microscopes' calibration procedure relies on the petroleum engineers' ability to determine what is acceptably sharp. The calibration procedure of both microscopes consists of three main parameters: THV, ESV/FRV, and depth of field, all of which must be manually tuned in order to measure the single-particle properties accurately. A calibration procedure was determined as a solution to keep a consistent procedure for calibrating the microscope. The

calibration procedure results showed high accuracy in measuring three different known particle sizes in a validation experiment, indicating a useful calibration procedure.

To evaluate both microscopes' performance to measure OiW concentrations in steady-state and real-time, oil was selected as a verification object as the fluorescence-based monitor can benchmark the microscopes ability to measure concentrations. Both measurements showed reasonable results in relation to the fluorescence-based monitor and the expected OiW concentration. A trailing moving average window of 1min was applied to evaluate both microscopes ability to measure the OiW concentration in real-time. Although depending on the plant's dynamics, it can be discussed whether the moving average window should be extended to smooth the continuous measurement even further. The errors related to the statistical representation of a particle size distribution were presented. A mathematical procedure proposed by Masuda and Iinoya [113] was used to determine the minimum number of particles that must be counted within a defined confidence interval and the amount of error allowed. An example was given of how long time it will take to obtain a satisfying particle size distribution by preferring a 95% of the data to be included within different relative errors.

Chapter 4.   Online Monitoring of Total Suspended Solids

Chapter 5

Graphical Interface on Industrial Software

Having a good understanding of the uncertainties associated with the online monitors would improve their industrial acceptance. Even though proper calibration is vitally important to ensure reliable measurement, presenting the data to an operator in an appropriate manner is at least as important. The traditional visualization approach is usually based on the assumption that calibration data are exact and, thus, the visual representation of uncertainty is ignored [115]. It is essential to convey the uncertainty in the right format in order for an operator to swiftly comprehend the process's current state, which can range from simple text to dynamic graphical representations, as discussed in Paper E. It is necessary to determine how best to present the data to an operator when there are uncertainties related to the data and how different representations can affect users' understanding of the uncertainty, all of which can affect the operator's decisions and actions. Inaccurate interpretation of the data can potentially lead to costly failures. According to Abnormal Situation Management (ASM) consortium, abnormal events cause $(3-8)\%$ production loss of the plant's capacity, where 42% of those are due to human incidents (operators), 36% are due to degradation and failures of equipment, and 22% are due to process operation [116]. $(3-8)\%$ production is equivalent to $\$(45-120)$ million lost annually in oil production in the Danish sector alone, with the assumption of a crude oil price at \$41 per barrel of oil, and the annual production of oil in 2019 of $5.851 \cdot 10^6 \mathrm{m}^3$ [9, 116]. Based on these aspects, it is essential to consider what should be displayed to the operator and how it should be displayed in order to strengthen the operator's comprehension of the system state. Operators can miss out on important information due to the large number of data streams flooding the operator's screens [117]. Therefore, suggestions are given to emphasize the "human factors" when designing HMI software for the

operators [117]. The HMI software is the graphical interface of the supervisory control and data acquisition (SCADA) system used offshore, on which the operator controls the process. For a simple system with a well-defined operating point, the task of identifying abnormal situations might be easy. However, for a complex interdependent system, where the optimal operating point varies due to uncontrollable inputs, the situation could be much harder to interpret [52]. In addition to incorporating water quality monitors into the decision support solution, either manually or through control feedback information, the data's presentation and interpretation must be evaluated. The water quality monitors should output accurately information and easy to interpret for an operator before having the potential to improve product quality, increase production, or lower OPEX, as mentioned in Sec. 1.1.

5.1 Human Machine Interface: a Case Study

Paper E presents a case study to compare MPC to conventional proportional, integral, and derivative (PID) control on the pilot-plant facility, as shown in Fig. 3.1. The case study of the control solution comparison uses the four configuration sections, as shown in Fig. 3.2: support, pipeline riser, separator, and hydrocyclone to execute the experiment. A design approach is given for displaying and explaining the control for the operators. This is based on uniting the fast prototyping capability of Simulink Real-Time with an industrial graphical interface HMI software with OPC as the central node in the communication system. The selection of establishing a connection between Simulink Real-Time and HMI is the basis of experimental execution that is particularly useful when developers aim to increase the readiness level of new solutions.
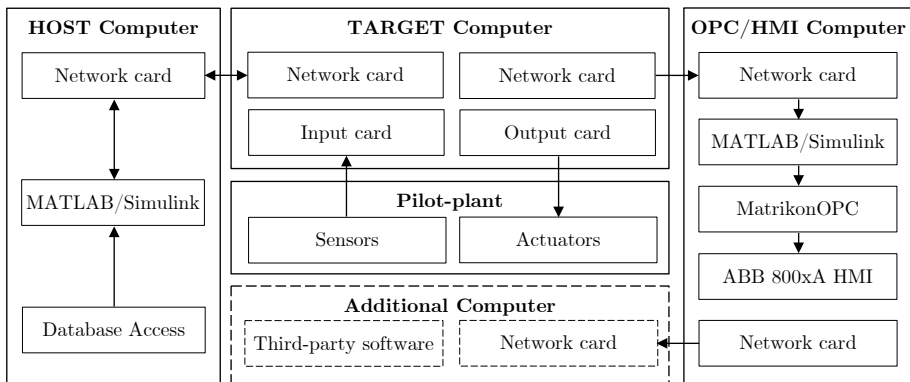
Fig. 5.1: The established connection between different computing systems of the pilot-plant. Each block represents a physical computer dedicated to the pilot-plant with its respective software and hardware. Figure is modified from Paper E.

Fig. 5.1 shows the connection of three dedicated computers and an optional connection to an additional computer if a third-party software should be evaluated.

Although this thesis does not cover the design of new offshore control solutions in the oil and gas sector, the case study was particularly useful at the time to investigate the potential of the communication establishment between Simulink Real-Time and the HMI software. The design solution of the MPC algorithm used for this case study has been published by Hansen et al. [55]. The graphical interface presentation of MPC algorithm is presented in Fig. 5.2. The PID control operation was ghosted on the MPC operation's presentation to evaluate their different performance under the exact same operating conditions of the pilot-plant facility. By comparing the visual design of the data presented in the HMI software shown in Fig. 5.2, and the image in Fig. 3.1 showing the Simulink Real-Time interface, it is much more challenging to comprehend the Simulink Real-Time interface, especially if larger quantities of data must be evaluated at the same time.

Based on the MPC case study, it will be beneficial to evaluate how to present the data from the quality monitors presented in this thesis. Especially if a third-party software connection through, e.g., OPC is not an option at a platform offshore, information must be delivered as signals to the PLC as part of the SCADA system. Although OiW concentration is not troublesome to deliver as a signal, delivering representative particle sizes within each class in continuous statistical batches can be more problematic.

5.2 Conclusion

The scaled pilot-plant provides a suitable testing environment for developing new features to the upstream offshore oil and gas industry, as it is capable of running different sections of the plant in series or bypass sections. To further mimic industrial practice, the plant's data were expanded by establishing a connection between Simulink Real-Time (academic used software) and ABB 800xA HMI software (industrial used software). The proposed connection can increase the adoption speed of new technological approaches by lowering the gap between academic research and industrial implementation when creating new solutions with a high innovation rate.

To demonstrate the connection functionality, a case study was carried out by investigating how to improve the operators' comprehension of the MPC procedure and how different controllers' performance can be presented in real-time by ghosting previous experiments. Although this work does not directly concern online quality monitoring, the data's presentation and interpretation can be investigated in an industrial used software in a safe testing facility.
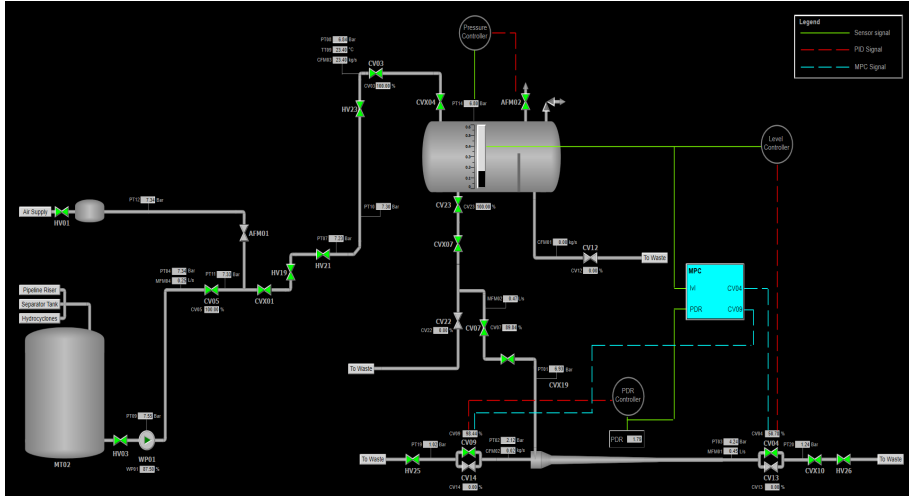
(a)
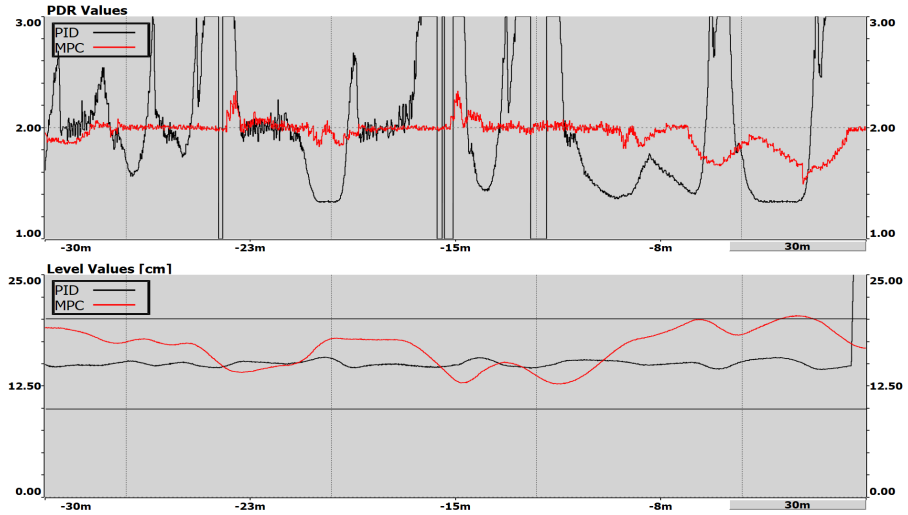
(b)

Fig. 5.2: The industrial used HMI interface: (**a**) shows the graphical design of the pilot-plant. (**b**) shows an example of a trend curve that can be visualized in the HMI software. In this particular example, the trend curve shows the level within the separator tank and the PDR of the hydrocylone with two different control algorithms: MPC and PID. Figures are from Paper E

Chapter 6

Closing Remarks

With growing requirements for increasing operational efficiency and environmental sustainability, the application use of online water quality monitors will increase in the years to come, whether it is in the water industry to enhance the drinking water quality or wastewater disposal or in the oil and gas industry to enhance the produced water discharge or to reinject the produced water into the reservoir. In this thesis, the challenges associated with online water quality measurements in the IWT process are addressed, emphasizing OiW concentration and TSS measurements. The thesis aims to increase the awareness of online monitors for the offshore oil and gas industry and highlights the challenges within online quality monitors. The main contributions of this work can be summarized to:

- An extensive literature review on what IW quality characteristics can be considered as TSS, which affects the reservoir's economic lifetime. Furthermore, no similar review study has discussed the complexity of selecting quality monitors that can reliably measure suspended solids' sizes and concentrations in an IWT process. Online Microscopy analyzers were deemed the best candidates to measure suspended solids based on the review study.

- Uncertainty analysis and calibration evaluations of online fluorescence-based monitors. The evaluated online fluorescence-based monitors showed a high reproducibility between each other using a weighted least square method to execute the calibration procedure. The performance of measuring OiW concentration with the fluorescence-based monitor showed promising results. However, the fluorescence-based monitor is highly sensitive to changes in the oil's aromatic content, which is a considerable limitation that will affect the fluorescence-based monitor's performance when oil is produced from different production wells.

- Calibration and performance evaluations of online microscopy analyzers. The evaluated online microscopy analyzers showed promising results in measuring particle sizes and concentrations. Furthermore, they were reasonable to measure OiW concentrations in both steady-state and real-time by using a trailing moving average window.

- Defined methodology to integrate and test novel process solutions with industrial used software. An industrial used software program was established to expand the in-house pilot-plant's data information to mimic the industrial practice of newly developed features, which includes presentation of water quality data.

Based on the literature review proposed in Paper A, the type of IW characteristics that can affect the increment of TSS concentration was comprehensively investigated, and the formation damage mechanism it can advance. While controlling water quality in the IWT process has been highlighted as the main key to increase the useful lifetime of the reservoir in several studies, most of these studies state that formation damage can be prevented or reduced, but they rely on the hypothesis that online accurate measurements are available.

In the second part of the review proposed in Paper A, a discrimination process was executed for petroleum engineers to reduce the number of choices when selecting instruments to measure TSS' quantity and size distribution in their IWT process. Measuring the single-particle properties with an online microscopy analyzer was concluded to be the best candidate to achieve TSS measurements in an offshore IWT process. To measure both OiW concentrations and single-particle properties online, each measurement type's advantages and limitations were divided into three papers, where Paper B and C cover evaluation of a fluorescence-based monitor to measure OiW concentration, and Paper D investigated two types of microscopy analyzers for measuring TSS. Four identical fluorescence-based monitors were examined in Paper C to estimate the OiW concentration. The calibration method of the OiW monitor revealed that WLS yields a higher reproducibility between the four monitors than the OLS method. Furthermore, an uncertainty analysis of sampling, calibration of the instrument, OiW concentration measurements with the reference method, and different variations in the process parameters were explored. Paper B proposed the application use of quality monitors on an offshore production treatment process for measuring the separation efficiency of a hydrocyclone. The use of fluorescence-based monitors to measure the separation efficiency of a deoiling hydrocyclone showed promising results capable of accurately measuring OiW concentrations under certain conditions. Paper D revealed that two types of online microscopy analyzers were able to measure known particle sizes and concentrations with high accuracy. Further examination of their ability to classify several different particles is necessary. Lastly, an expansion of the pilot-plant's interface was established in Paper E by integrating an industrial

used software program to be able to improve the presentation of the data from new features such as online water quality monitors. In conclusion, as there is no consensus on which method provides the most reliable estimate of TSS in an IWT process, the author has put a considerable effort into investigating what measurement type will be most promising for measuring TSS.

By acknowledging the uncertainty related to measuring the different kinds of water qualities in an IWT facility, future work may take into account these uncertainties when new water quality monitors need to be investigated or developed monitors need improvements. Accurate water analysis is essential to identify process changes and promotes sophisticated process optimization and control to be implemented into any water system.

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