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Angelo Camerlenghi  
Char-Shine Liu  
*Editors*

# World Atlas of Submarine Gas Hydrates in Continental Margins

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Editors

# World Atlas of Submarine Gas Hydrates in Continental Margins

 Springer

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Cover image: Giant blow-out craters and active gas flares from the shallow (< 350m) Barents Sea floor document massive reactions of shallow gas hydrate provinces to ice sheet thinning and retreat during the postglacial. Courtesy of Andreia Plaza Faverola, CAGE—Centre for Arctic Gas Hydrate, Environment and Climate, UiT- The Arctic University of Norway, Tromsø, NO.

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## Preface

The *World Atlas of Submarine Gas Hydrates in Continental Margins* is a comprehensive global compilation of geophysical evidence for the presence of natural gas hydrates in the seafloor. Gas hydrates represent a major carbon reservoir in the Earth system that traps vast amounts of methane below the seafloor. Hydrates form through the capture of gas molecules in water molecule cages when there is sufficient water and free gas and if temperature and pressure conditions are met. Favorable conditions for gas hydrate formation can usually be found beyond the shelf edge and within the top few hundred meters below the seafloor. Because the kinetics of gas hydrate formation and dissociation are fast compared to many other geologic processes, this reservoir is dynamic and sensitive to climatic and tectonic perturbations. Thus, the gradual warming of the seafloor may destabilize gas hydrates, leading to gas blowouts and possible destabilization of the seafloor. The process of mapping the distribution of gas hydrates is therefore critical for the evaluation of offshore geohazards. As methane is the main component of natural gas, the methane in gas hydrates is also considered a future energy source, especially for countries that lack conventional hydrocarbon reservoirs. In the absence of photosynthesis, bacteria that feed on methane are one base of the food chain in the deep ocean. Gas hydrate formation, dissociation, and bacterial activity are important modulators for the flux of methane to the seafloor. For all these reasons, gas hydrate research has played a prominent role in the field of marine geology and microbiology over the past three decades.

As the most striking geophysical observation linked to gas hydrates is the bottom simulating reflector (BSR), we initially considered calling this book “The Atlas of Bottom Simulating Reflectors.” A gas hydrate-related BSR shows a phase reversal compared to that of the seafloor. It is caused by an abrupt change in acoustic impedance at the boundary between the gas hydrate-bearing sediments above the BSR and the sediments containing free gas below. The BSR thus represents the base of the gas hydrate stability zone (GHSZ) and can be used to infer subseafloor temperature and pressure conditions as well as the presence or absence of gas hydrates and free gas. The atlas fills a major gap in the literature of geophysical exploration through the compilation of typical shapes and seismic expressions for almost all ocean areas where gas hydrates have been reported or suspected. This covers vastly different geological settings, including volcanic and non-volcanic passive margins as well as oceanic and continental subduction zones. By summarizing the main findings for each of these areas, the book both provides an overview of the occurrence of gas hydrate-related BSRs in different geological settings and with different types of geophysical data. It also provides new insight into the processes and time scales that affect gas hydrates.

Observations of BSRs in 2D and 3D seismic reflection data, combined with detailed analysis of P- and S-wave velocity and attenuation, electrical resistivity imaging, and gas hydrate stability zone (GHSZ) modeling, aid researchers in both academia and the hydrocarbon industry in understanding the potential extent and volume of gas hydrates in a wide range of tectonic settings on continental margins. Sub-seabed imaging techniques provide insight into the controlling mechanisms for the distribution and migration of gas as it enters and moves through the gas hydrate stability zone. Repeated imaging at the same site uncovers new details regarding the dynamic behavior of these systems. Over the past few decades, drilling campaigns such as the Deep Sea Drilling Project (DSDP), the Ocean Drilling program

(ODP), and the International Ocean Drilling/Discovery program (IODP) have allowed for the direct sampling of gas hydrates. They have been instrumental in constraining the geological and physical boundary conditions necessary for the formation of natural gas hydrates in continental margins and lakes. This volume summarizes these results and discusses the geophysical observations in this context.

The *World Atlas of Submarine Gas Hydrates in Continental Margins* is aimed at students, researchers, governmental organizations, and professionals from the hydrocarbon industry. Some familiarity with seismic data and some basic understanding of geological and tectonic processes will be required to get the most out of this volume. Apart from presenting a short overview of gas hydrate science (e.g., geology, geophysics, modeling), its main aim is to provide a global perspective on the variable geophysical observations related to gas hydrates in different parts of the world. With comprehensive references to key papers covering each location, it should also provide a good starting point for those who are new to a particular gas hydrate province. It will also provide ample teaching material for classes in marine geology and geophysics.

The atlas consists of fourteen parts containing a total of 43 peer-reviewed articles written by esteemed researchers from universities and government agencies around the world. The articles provide both new data and reviews of previously published data. Geophysical interpretations are discussed in the context of drilling and coring results when possible to ground truth the geophysical findings. The majority of contributions describe and discuss geophysical data from gas hydrate systems worldwide. These contributions are organized by geographic area (see map) and may serve as a reference for documenting future changes. Future researchers may use this comprehensive compilation of gas hydrate stability zone data to further investigate questions such as: What is the gas hydrate inventory for active and passive margins? Where are the most climatically sensitive gas hydrate reservoirs on Earth and how fast can they respond to geological and oceanographic perturbations? Which gas hydrate occurrences should be considered geological hazards? How do the geophysical characteristics of BSRs differ in various geological settings?

Each of the 14 parts of the atlas cover topics of international interest in documenting gas hydrates:

- I A History of Gas Hydrate Research (Chaps. 1–3)
- II Gas Hydrate Fundamentals (Chaps. 4–6)
- III Gas Hydrate Drilling for Research and National Resources (Chaps. 7–16)
- IV Arctic (Chaps. 17–21)
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We are grateful to all our colleagues who contributed to this atlas documenting the characteristics of gas hydrate systems on continental margins.

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 Taipei, Taiwan

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The World Atlas of Submarine Gas Hydrates in Continental Margins documents the high level of international research efforts necessary to understand the global occurrences of natural gas hydrates. Substantial knowledge increases about where gas hydrates lay in different geological environments have been made possible through decades of funding and a culmination of several years of dedicated geophysical exploration and gas hydrate sampling.

We are grateful to all our colleagues who contributed to this atlas documenting the characteristics of gas hydrate systems on continental margins. We thank those from the United States Geological Survey for encouraging us to compile this book, and we thank the Norwegian Research Council for providing financial support for this effort through funding the Centre of Excellence for Arctic Gas Hydrate, Climate and Environment (CAGE) at UiT—The Arctic University of Norway (grant 223259). We thank Jessica Green, a science writer at UiT funded by CAGE, for her help with language cleansing and styling guidelines. We also wish to acknowledge and thank the numerous national and multinational funding sources that enabled the geophysical expeditions that were necessary to acquire the data presented in this book.

Jürgen Mienert

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### About the Editors



**Jürgen Mienert** is a Professor Emeritus for Applied Geophysics and Arctic Marine Geology at UiT—The Arctic University of Norway, Tromsø, Norway. He chaired the Department of Geology from 2008 to 2012, before he received an award for a Centre of Excellence by the Norwegian Research Council in 2012. He was Founding Director of this Centre for Arctic Gas Hydrate, Environment and Climate—CAGE—from 2013 to 2017. His research focuses on understanding the rate at which rising ocean temperatures can destabilize shallow, Arctic methane hydrate reservoirs leading to geohazards and methane release to the ocean and atmosphere. Jürgen received his Ph.D. in marine geoscience from the Christian-Albrechts University (CAU) at Kiel, Germany (1985). He was awarded a DAAD fellowship for studying at LDGO—Lamont-Doherty Observatory of Columbia University, New York (1981–1982) and was awarded a post-doctoral position at WHOI—Woods Hole Oceanographic Institution, Woods Hole, USA (1985–1988). He returned to Kiel in 1988 to join the newly established Centre for Marine Geosciences—GEOMAR—at the CAU, Kiel, before he moved to the Arctic University of Norway in Tromsø (1998). His collaboration with teams from Russia, USA, Canada, and Europe on polar shelf and slope environments facilitated active cooperation among hydrocarbon companies, technology providers, and Arctic research groups. His many sea expeditions included contributions to the Norwegian deepwater gas field “Ormen Lange” investigation, dives with Russian submersibles MIR from RV Keldysh (1998) to the deep ocean floor at the giant submarine “Storegga slide” on the Mid-Norwegian Margin and to natural gas release sites along the Barents-Svalbard Margins, which provided an indispensable guide for methane hydrate studies. His international research encompassed stays at Scripps Institution of Oceanography, USA (2002–2003), IFREMER—L’Institut Français de Recherche pour l’Exploitation de la Mer, FR (2007–2008), MBARI—Monterey Bay Aquarium Research Institute, USA (2017–2018), and OGS- Istituto Nazionale di Oceanografica e di Geofisica Sperimentale, IT (2018–2019). Jürgen has authored and co-authored more than 200 papers in scientific journals and special volumes.



**Christian Berndt** is Professor of Marine Geophysics at GEOMAR Helmholtz Centre for Ocean Research Kiel. He has a background in active source seismology. He has been working for the past 20 years on various aspects of fluid migration in sediment basins. These include the role of gas hydrates in the Earth system, hydrothermal systems caused by magmatic intrusions, and the links between pore fluids and slope stability. He held academic positions in the U.S.A., Norway, and the United Kingdom and has led or participated in more than 30 research expeditions all over the world.



**Anne Tréhu** is a Professor of Geophysics in the College of Earth Ocean and Atmospheric Sciences at Oregon State University. She obtained her Ph.D. from the MIT/WHOI Joint Program in 1982, where she specialized in ocean bottom seismometry and its application to seismicity and structure of plate boundaries. She has been interested in gas hydrates since she was introduced to them by Bill Dillon when she worked at the United States Geological Survey in Woods Hole, Massachusetts (1982–1987). She has been at Oregon State University since 1987, where her work on hydrates dovetails with her parallel interest in structures associated with subduction zone earthquakes since accretionary wedges provide a mechanism for methane to generate seafloor cold seeps and rich hydrate deposits. In 2004, she was Co-Chief Scientist of IODP Leg 204, which was dedicated to understanding the distribution and dynamics of gas hydrates associated with a structure named Hydrate Ridge on the continental margin offshore Oregon. She has published ~150 articles, with approximately one-third focused on gas hydrates, and participated in over 40 field expeditions, including one dive in Alvin and five expeditions using ROVs to explore and sample cold seeps and hydrates.



**Angelo Camerlenghi** is Director of the “Geophysics” Research Section of the National Institute of Oceanography and Applied Geophysics (OGS) and Associate Professor of Marine Geology at the University of Trieste. He holds a Master of Science in Geological Oceanography from Texas A&M University, College Station TX (1988) and a Ph.D. in Earth Sciences from the University of Milano (1991). From 2004 to 2011, he was ICREA Research Professor at the University of Barcelona. His research is on ocean margins geology and geophysics, with focus on polar margins, submarine geohazards, and manifestations of formation fluids circulation in marine sediments. His research is tightly related to scientific ocean drilling. He has studied the geophysical evidence of marine gas hydrates on the South Shetland Margin, Cascadia Slope, West Svalbard, and the mud volcano belt of the Eastern Mediterranean. His research is currently on the Mediterranean salt giant.



**Char-Shine Liu** is a Senior Researcher and CEO of the Ocean Center, National Taiwan University, Taipei, Taiwan. He was the Director of this Center from 2016 to January 2018 when he retired from faculty position at the Institute of Oceanography, National Taiwan University, where he was a Professor since 1994. During his Professor tenure, he also served as Director of the National Center for Ocean Research from 2003 to 2006 and Chairman of the Taiwan SCOR Committee from 2004 to 2009, among many other roles in his career. Char-Shine received his Ph.D. in Earth Science from the Scripps Institution of Oceanography, University of California, San Diego, USA in 1983. He did a 2-year (1983–1985) postdoc work at the Cornell University, New York, USA, followed by a 3-year stint as a geophysicist at then Standard Oil Company (now BP) in Dallas, Texas, USA, before returning to Taiwan in 1988 and took an Associate Professor position at the Institute of Oceanography, National Taiwan University. He has broad research interests from active tectonics of the Taiwan arc-continent collision to marine seismic exploration, seafloor surveying, and gas hydrate investigation. Char-Shine has collaborated with colleagues from USA, France, Germany, and Japan on various research topics from tectonics of Taiwan to gas hydrate study. He has led geophysical investigation of the gas hydrate field offshore southwestern Taiwan since 2004, and has authored and co-authored more than 140 papers in scientific journals and special volumes.

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**Part I**

**A History of Gas Hydrate Research**



# Gas Hydrate Research: From the Laboratory to the Pipeline

1

Jose G. Delgado-Linares and Carolyn A. Koh

## Abstract

Gas hydrates have been the subject of intensive research over the past several decades. Complications created by hydrate formation and the plugging of gas and oil flowlines has been the main driver in the development of predictive models based on experimental observations. The multiscale approach discussed in this article is based on many years of systematic investigations; phenomena related to gas hydrate formation are considered at the microscale to the macroscale. The processes surrounding hydrate nucleation, growth and agglomeration are critical to designing strategies for hydrate plug avoidance and management. An overview of the main key experimental techniques used in hydrate research will be presented, with special emphasis on how those techniques may provide valuable input in improving integrated hydrate models in different flow assurance scenarios.

## 1.1 General Aspects

Gas hydrates are solid inclusion compounds in which a hydrogen-bounded water network (the host) encapsulates small gas molecules (the guests). Common varieties of hydrate guests, also known as ‘formers’, include small hydrocarbons (e.g. methane, ethane), noble gases (e.g. xenon), diatomic gases (e.g. nitrogen), and fluorinated compounds (e.g.  $\text{CH}_2\text{FCF}_3$ ) (Sloan and Koh 2007).

Although formation conditions are specific to a particular guest, hydrates are always stable at high pressure and low temperature (Sloan and Koh 2007). On a microscopic level, hydrates can form three different crystalline structures; these are known as Structure I (sI), Structure II (sII) and

Structure H (sH). Structures sI and sII are of special interest as they can trap the small gas molecules found in both natural and industrial systems. In all three of the crystal configurations, the hydrogen-bounded water molecules form molecular cages, as shown in Fig. 1.1. The  $5^{12}$  cage is considered a basic building block of these structures (Giavarini and Hester 2011; Jeffrey 1984; Koh et al. 2011; Warrier et al. 2016).

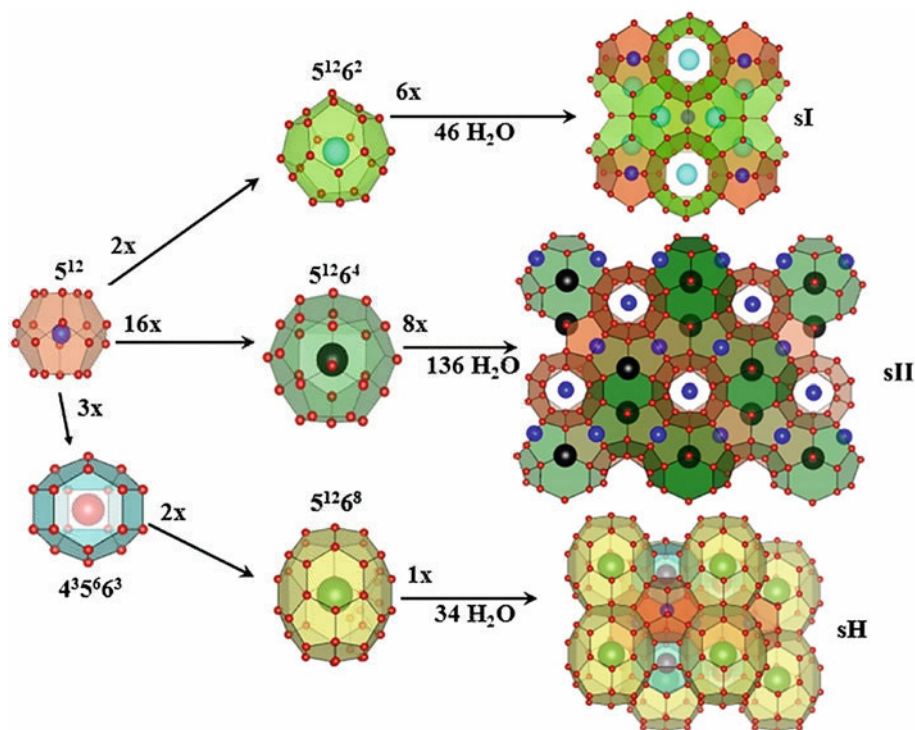
It is the size of the guest that determines which of the three structures will be created. Thus, gases smaller than 6 Å (e.g. methane, ethane, and carbon dioxide) induce the formation of Structure I, molecules between 6–7 Å (e.g. propane and isobutane) form Structure II, and molecules larger than 7 Å (e.g. iso-pentane, combined with a small guest such as methane) promote the formation of Structure H (Sloan and Koh 2007).

The increasing interest in gas hydrates is largely due to their potential as an energy resource. They are a non-conventional fossil fuel able to store up to 164 m<sup>3</sup> of methane gas for each cubic meter of hydrate, which is a high value of energy density compared to conventional fuel sources (Demirbas 2010). Another advantage of hydrates as a prospective fuel source is their abundance in nature, representing over half of the total reserves of organic carbon on Earth (Mahajan et al. 2007). Further research on natural methane hydrates motivated by implications for climate change, submarine geohazards, and geo-microbiological processes at the seafloor exist but are outside the scope of this article.

Gas hydrates have potential application in several technological areas, as shown in Table 1.1. In some cases, gas hydrate occurrence is not only desirable but critical in accomplishing a process goal (e.g. gas separation, desalination). On the other hand, the plugging of flowlines by gas hydrates in conventional offshore oil/gas operations represents a flow assurance problem, which can reduce or even stop the hydrocarbon flow. A similar problem can also occur in the extraction of gas from naturally occurring hydrate sources. Thus, significant effort and expense have been put

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**Fig. 1.1** Main gas hydrate structures. Number of cages and water molecules per unit is indicated (reproduced with license granted by AIP Publishing from Warriar et al. 2016)



**Table 1.1** Summary of hydrate applications

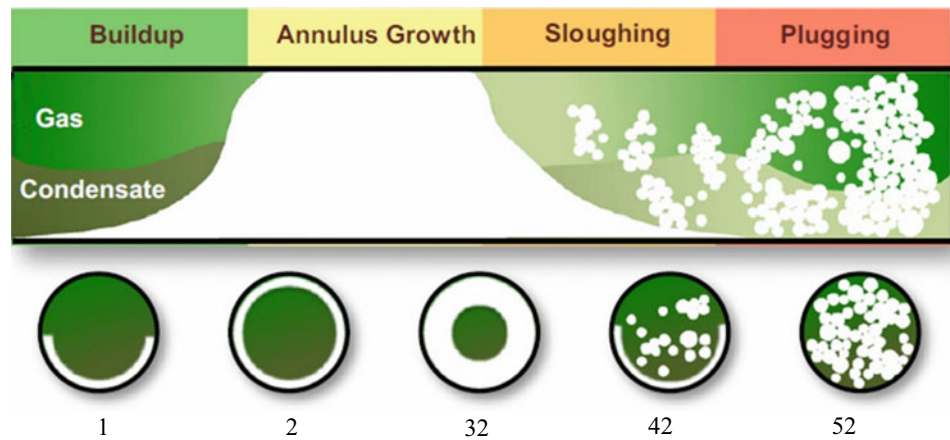
Hydrate application	Guest/structure type	Focus areas	References
Hydrates in nature	Methane/sI (biogenic); natural gas mixtures (thermogenic)	Resource exploration and assessment; gas production; geomechanics; Environmental impacts	Boswell et al. (2020) Collett (2019) Moridis et al. (2018) Ruppel and Kessler (2017)
Flow assurance	Typically sII, natural gas mixtures	Thermodynamics; LDHI-KHIs & AAs; Non-plugging oils; reaction limitations; multiphase flow	Sloan and Koh (2007) Kelland (2016) Sjöblom et al. (2010) Turner and Grasso (2017) Wang et al. (2018)
Energy storage	sI (methane); sII (hydrogen and natural gas)	Stability/capacity—NG Stability—H <sub>2</sub> Rapid formation	Stern et al. (2003) Florusse et al. (2004) Veluswamy et al. (2016)
Gas separation	sI, sII	Gas selectivity	Warriar et al. (2018)
Desalination	sI, sII	Salt exclusion; crystal morphology	Khan et al. (2019)

into effect to develop practical strategies for hydrate avoidance and management in production systems (Creek 2012; Sloan and Bloys 2000).

Beginning in 1810 with the discovery of gas hydrates by Sir Humphrey Davy (1811), the evolution of its research has been guided by several critical events, including detection of the formations in pipelines (1934) and later in natural environments (1965). The complications that gas hydrates

create in the flow assurance of pipelines has been a driving force in the study of phenomena related to hydrate behavior. In this sense, a cross-disciplinary approach based on microscopic and macroscopic observations has been used alongside the development and implementation of predictive tools, giving rise to major advances in hydrate science. Many of these advances can be applied both within pipelines and in the natural environment (Sloan 2004).

**Fig. 1.2** Conceptual picture of hydrate formation and plugging in gas-dominated systems. The green color represents the gas and the white color represents the hydrates (modified from Lingelem et al. 1994 and reproduced with license granted by Elsevier from Zerpa et al. 2012)



One of the most important factors contributing to the formation of hydrates in flowlines is the relative amount of each phase present (e.g. gas, oil, or water). Thus, the mechanism for hydrate formation in oil-dominated systems is different from that of gas-dominated systems, even if some phenomena are common in both systems. Most research on the effect of gas hydrates on flow assurance in pipelines use large-scale observations as a starting point (Sloan 2004). There is a necessity to perform experimental work on a smaller scale using equipment and experimental techniques in the laboratory to obtain more detailed information on specific phenomena (e.g. viscosity, adhesion etc.) related to gas hydrates.

As mentioned, gas hydrates are commonly known to form within both gas- and oil-dominated systems, creating blockages that lead to flow problems. The conceptual model for hydrate formation and plugging in gas-dominated systems is shown in Fig. 1.2. It can be summarized as follows: (1) hydrates start to nucleate at the pipe surface, (2) after nucleation, hydrates grow rapidly and cover the circumference of the pipe, (3) the effective diameter of the line is reduced because of hydrate growth, (4) part of the deposit can detach from the wall (sloughing) due to fluid shear, and (5) hydrate particles can accumulate in other parts of the line

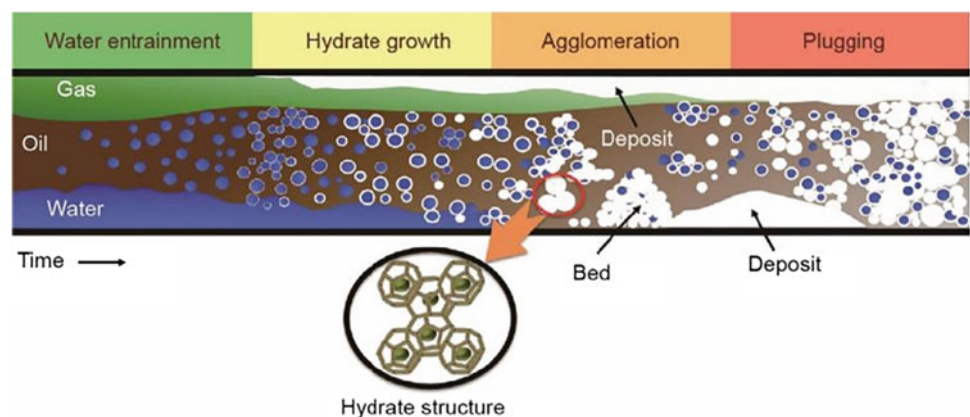
(e.g. flow restriction) and lead the system to jam (Lingelem et al. 1994; Sloan et al. 2010; Sum et al. 2012; Zerpa et al. 2012).

Figure 1.3 depicts the conceptual model of hydrate plugging in an oil-dominated system (Majid et al. 2018). The relatively high proportion of oil and water typically makes the entrainment and emulsification of water into the oil phase a determinant step for hydrate formation. In this case, a hydrate film grows quickly around the water droplets, creating hydrate shells (5–30  $\mu\text{m}$  thick) with a water core; finally, the hydrate-coated particles agglomerate to plug the flowline (Sloan et al. 2009, 2010).

Figure 1.3 shows that phenomena such as viscosification, film growth, deposition, bedding, and jamming can occur simultaneously. In contrast to gas-dominated systems (shown in Fig. 1.2), the oil chemistry of oil-dominated systems is a primary factor that can assist or restrict hydrate agglomeration and plugging (Costa Salmin et al. 2019; Fadnes 1996; Leporcher et al. 1998; Sjöblom et al. 2010; Zerpa et al. 2011).

To build and quantify conceptual models like those in Figs. 1.2 and 1.3, many observations and measurements are required in different experimental setups and at different

**Fig. 1.3** Conceptual picture of hydrate formation and plugging in an oil-dominated system (reproduced with license granted by Elsevier from Majid et al. 2018)





scales; furthermore, phenomena such as hydrate nucleation, growth, inter-particle adhesion, wettability and jamming must also be considered. Once the microscopic phenomenology is developed, the next step consists of performing experiments at a pilot scale to obtain information on the system behavior in more realistic conditions before advancing towards field applications. It is important to keep in mind that an increase in the experimental scale also generates an increase in the volume of fluids required, while the control of experimental conditions diminishes. The final goal of gas hydrate research in flow assurance is to build a comprehensive model capable of predicting hydrate formation and plugging in certain conditions so that hydrate prevention/management strategies can be efficiently applied.

The main objective of this article is to present a general overview of the main experimental techniques and apparatuses used for hydrate research, with emphasis on the measurement principles, operation conditions and major outcomes.

## 1.2 Experimental Hydrate Research

### 1.2.1 Multiscale Approach

The formation of gas hydrates is a complex process that depends on many factors, including the types of phases present and the conditions under which those phases interact. While discussions presented here are limited to the flow assurance problems created by gas hydrates in flowlines, some generalization can be made to the formation of hydrates in other applications.

A hydrocarbon flowline is generally dominated by crude oil, water, gas and/or condensate. Thus, once pressure-temperature conditions are met, the potential for hydrate formation and subsequent plugging will primarily depend upon the relative amount of each phase present. Several other parameters may also play a part, such as hydrodynamic conditions (e.g. flow rate), viscosity, chemical composition of the fluids (e.g. gas composition, water salinity, natural surfactants in the oil, added chemicals, etc.), and flow patterns among others. The formation of hydrate blockages has been conceptualized in four main models: (1) oil-dominated, (2) gas-dominated, (3) gas condensate and (4) high-water cut systems (Sloan et al. 2010). All of these types of blockages are impacted by certain physical phenomena, ranging in scale from microscopic hydrate particle/film formation to macroscopic agglomeration and plugging.

Significant efforts have been made to develop practical tools capable of predicting hydrate formation. The general workflow can be summarized in three main steps: (1) small scale experiments, (2) pilot plant tests and (3) field applications and modelling. In general, laboratory experiments

require only a few micro- or milli-liters of sample material and allow for the study of physical phenomena in great detail under rigorous environmental control. When increasing the experimental scale towards pilot plant tests such as flow loop experiments, conditions become more realistic and the consumption of fluids and chemicals increases significantly; liters and even barrels of water, oil and gas are normally required to obtain datapoints.

Once the phenomenon is sufficiently understood, the next step is to test the physical/mathematical models developed at the pilot plant level on real-world field cases with the help of industrial companies. As a part of the field application, physical models are integrated with predictive tools to assess the risk of hydrate formation and plugging in real scenarios. For example, the predictive tool CSMHyK can be coupled with the transient multiphase flow simulator OLGA®, and the combination can then be used to predict hydrate formation and transportability in pipelines (Boxall et al. 2009; Zerpa et al. 2012).

Figure 1.4 is an illustrative summary of the multiscale approach used in hydrate research. It lists the main phenomena involved in hydrate formation and plugging and further identifies the equipment/techniques used at each scale; the brown text identifies factors applying exclusively to oil-dominated systems.

It is worth noting that the multiscale scheme presented in Fig. 1.4 may not only be used in gas hydrate formation studies but also as a practical tool to evaluate the efficiency of both natural and commercial chemicals in the management of gas hydrates in oil and gas flowlines (Dapena 2019; Hu 2019). The physics behind each experimental technique is the main factor to be considered in defining its specific application.

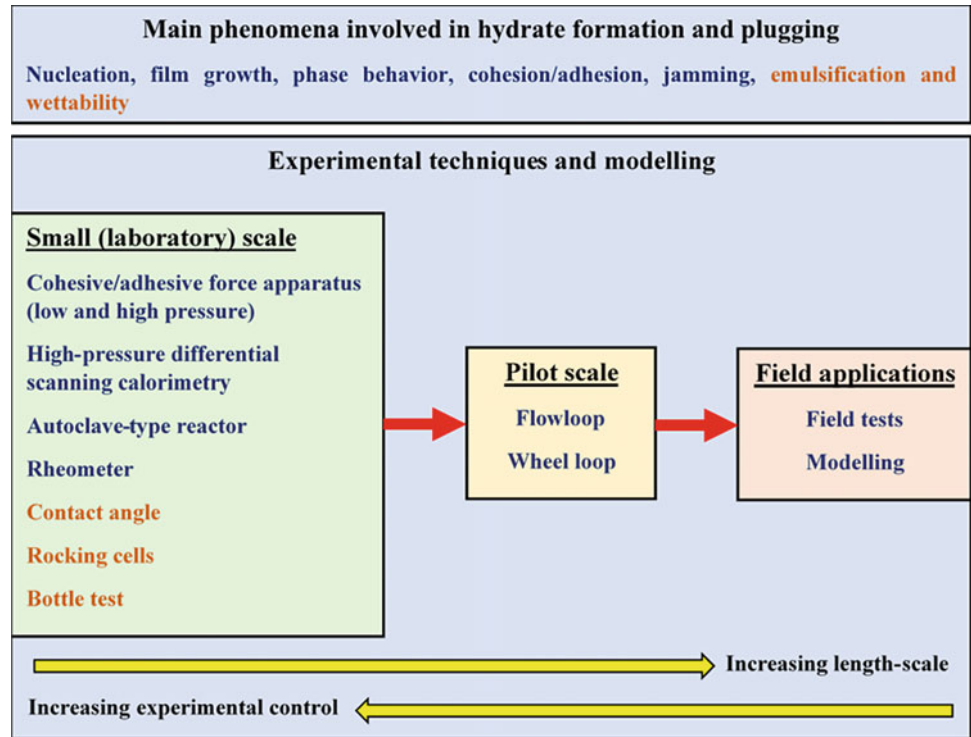
### 1.2.2 Overview of Experimental Techniques

The number of published works found on gas hydrates is enormous, implying a correspondingly high number of equipment/techniques applied in hydrate research, as shown elsewhere (Sloan and Koh 2007). Depending on the goal of each experiment, a setup is designed or adapted to collect a specific kind of data and the experimental conditions are fixed accordingly. Key experimental techniques given in Fig. 1.4 are briefly described, and their importance as a part of the conceptual models in Figs. 1.2 and 1.3 is highlighted.

#### 1.2.2.1 Small (Laboratory) Scale

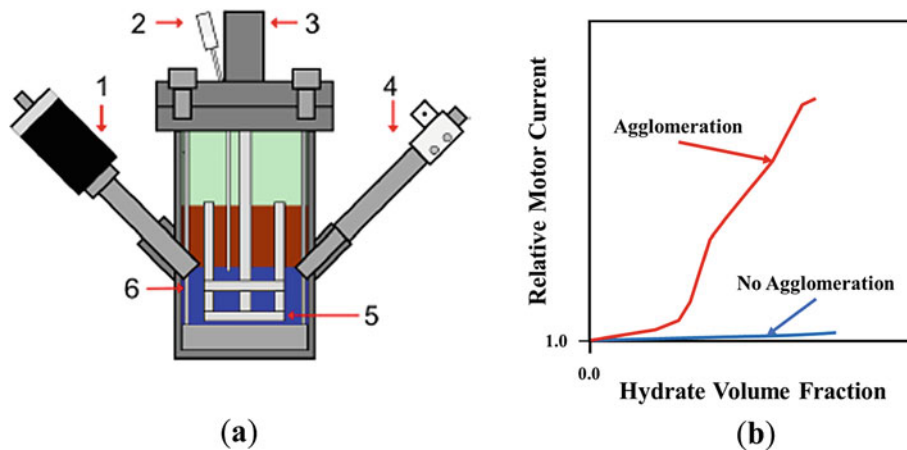
**Autoclave-type reactor.** The first technique covered is the use of batch reactors such as autoclave-type reactors/cells (Giavarini and Hester 2011). They are primarily used to determine pressure and temperature conditions as well as the kinetics under which hydrates are formed/dissociated. The cell, equipped with a stirrer, is connected to a gas reservoir

**Fig. 1.4** A multiscale approach for hydrate research. Techniques and phenomena appearing in brown apply only to oil-dominated systems



and placed in a thermal bath to reach the conditions necessary to form hydrates. Sensors are placed inside the cell to determine pressure and temperature, as well as optical devices such as Particle Video Microscope (PVM) and Focused Beam Reflectance Measurement (FBRM) probes, if available, to monitor the evolution of particle size over time (Costa Salmin 2019). Autoclaves can be designed to operate at pressures up to 5,000–10,000 psi (Sloan and Koh 2007), although the PVM/FBRM probes have an operation limit of around 1,000 psi. An example of this type of reactor and its main components is shown in Fig. 1.5a.

Autoclaves can be used to quantify the kinetics of hydrate formation in both oil-dominated systems as well as water/gas systems. They are also useful in evaluating the effectiveness of various hydrate inhibitors, such as thermodynamic hydrate inhibitors (THIs), kinetic hydrate inhibitors (KHIs) and anti-agglomerates (AAs). Further, they are capable of gathering information on the flowability properties of the hydrate slurry, namely its viscosity and particle size distribution (Akhfash et al. 2017; Chen et al. 2014; Majid et al. 2014; Salmin et al. 2017; Sun et al. 2015). Figure 1.5b shows a comparison between systems that display



**Fig. 1.5 a** Schematic of a high-pressure autoclave reactor. Main components: (1) Video probe (PVM), (2) thermocouple, (3) motor shaft (stirrer), (4) laser-scanning probe (FBRM), (5) impeller and (6) baffles

(Costa Salmin 2019). **b** Illustration of typical motor current variation as a function of hydrate volume fraction for systems with and without hydrate agglomeration

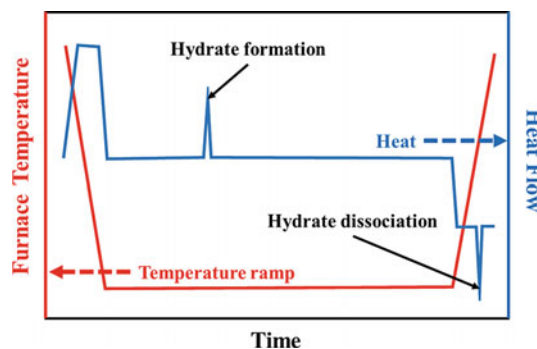
agglomeration versus systems that do not. The absolute motor current as it relates to system viscosity is illustrated as a function of the hydrate volume fraction, and it increases in systems where agglomeration is found.

#### High-pressure differential scanning calorimetry.

Another technique often used in hydrate laboratories is called differential scanning calorimetry (DSC), which is based on the measurement of the enthalpy variation as a function of temperature during heating and cooling cycles. The thermogram recorded by a DSC allows for the identification of phase transformations, such as hydrate formation and dissociation. These instruments are designed to operate at pressures of up to around 5,000 psi and at temperatures in the range of 230–400 K, with the advantage of using a very small amount of sample. Important information such as hydrate dissociation temperature, heat capacity, heat of dissociation, hydrate agglomeration and emulsion stability with and without hydrates may be obtained through this technique (Dalmazzone et al. 2003; 2009a, b; Delgado-Linares et al. 2013; Giavarini and Hester 2011; Lachance et al. 2008; Palermo et al. 2005; Sloan and Koh 2007).

A typical thermogram for a HP-DSC hydrate experiment is shown in Fig. 1.6, where temperature and heat flow as a function of time are plotted. The peaks in the heat flow trace correspond to hydrate formation and hydrate dissociation (Lachance 2008).

**Rheometer.** One of the most important properties considered when dealing with flow assurance problems is the viscosity of the liquid phase. An increase in the system's viscosity generally requires more vigorous pumping and increases the likelihood of plugging. Several studies have placed focus on evaluating the rheological behavior of hydrate suspensions, using flow loops and rheometers at low and high pressures (Delahaye et al. 2008; Majid et al. 2019; Siquin et al. 2004; Webb et al. 2012). The use of high-pressure rheometers has aided the development of some empirical and semi-empirical models to predict the viscosity



**Fig. 1.6** An illustration of a typical thermogram of a water-methane system indicating hydrate formation and dissociation

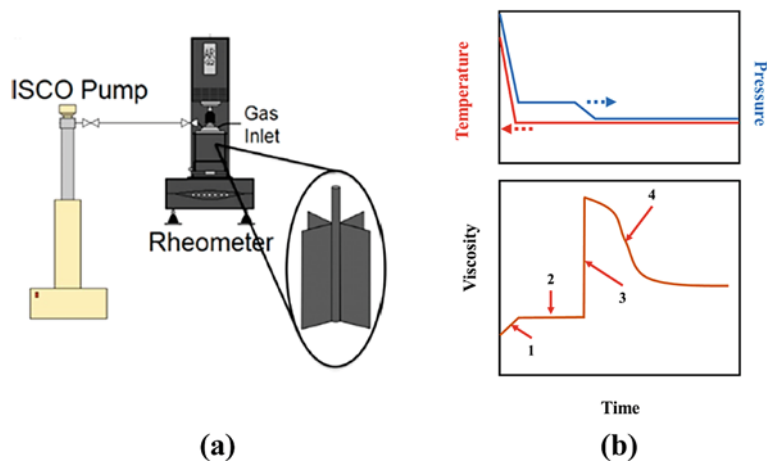
variations of emulsions and hydrate suspensions and slurries at different conditions of pressure and temperature (Camargo and Palermo 2002; Majid et al. 2017; Qin et al. 2018). Pioneering research in this field has explained the aggregation of hydrate particles in oil-dominated systems through the formation of inter-particle water bridges that generate attractive capillary forces (Camargo and Palermo 2002). An empirical equation used to predict the viscosity of stable water-in-crude oil emulsions has been recently proposed as a tool for analyzing the effect of hydrate particles on the viscosity of slurries (Majid et al. 2017).

Figure 1.7a is a schematic diagram of a high-pressure rheometer used in the study of hydrates. Figure 1.7b shows a typical viscosity-versus-time curve and the corresponding pressure and temperature profiles present for a system in which hydrates are formed in a water-in-oil emulsion. Four regions may be observed in this kind of system, as identified in the figure: (1) initial viscosity increase due to the cooling process, (2) viscosity remains constant, (3) viscosity rises suddenly and (4) viscosity decreases gradually.

The increase in the system viscosity in region 3 is the result of several combined factors, including the conversion of water droplets to hydrate particles, the depletion of

**Fig. 1.7 a** Schematic of high-pressure rheology (reproduced with license granted by the American Chemical Society from Majid et al. 2017).

**b** Illustration of typical temperature, pressure and viscosity profiles for hydrates formed in a water-in-oil emulsion



methane in the liquid phase, and the formation of capillary bridges between partially/fully converted hydrate particles. The viscosity reduction in region 4 may be explained through two main hypotheses. One is the re-saturation of the liquid phase with methane (gas former), reducing the viscosity of the hydrate suspension. The other involves the breakup and rearrangement of hydrate aggregates in the liquid phase (Majid et al. 2017; Webb et al. 2012).

**Cohesive/adhesive force apparatus.** As can be seen in Figs. 1.2 and 1.3, the aggregation of gas hydrate particles is a critical step in the plugging of pipelines. A micro-mechanical force (MMF) apparatus has been used to measure the interaction forces among hydrate particles and to quantify the interactions between hydrate particles and solid surfaces at ambient and high pressures (Aspenes et al. 2010a; Hu and Koh 2017; Lee and Sum 2015; Taylor et al. 2007; Yang et al. 2004). The experimental procedure for measuring hydrate interaction forces consists of 4 steps (Aman et al. 2012a; Hu and Koh 2017): (1) two water droplets are each attached to a glass fiber cantilever and converted to hydrate particles, (2) the particle on the top is moved against the bottom particle in order to provide a preload force for a specific period of time (3) the particle on top is raised at constant velocity, and (4) the movement is stopped when the particles are broken apart at a distance  $\Delta d$ . Hook's Law is then applied to calculate the cohesive force ( $F_a$ ), as follows:

$$F_a = k \Delta d \quad (1.1)$$

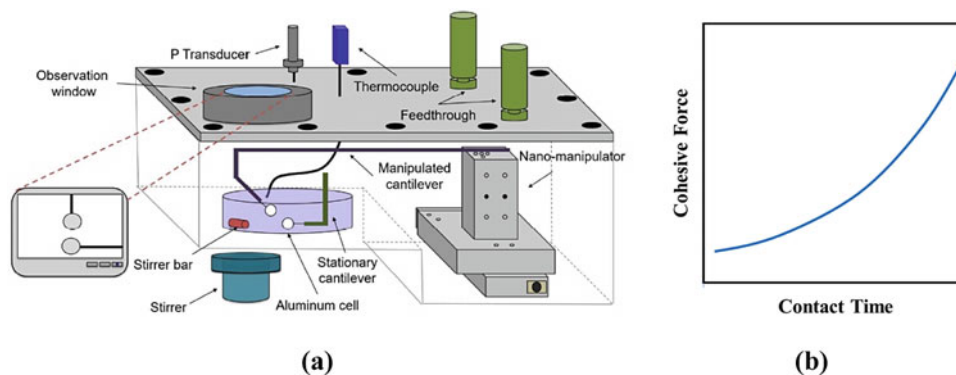
where  $k$  is spring constant of the glass fiber.

The MMF technique measures the impact of important operational parameters on the interaction of hydrate particles, such as subcooling, annealing time, contact time, and nature of dominant phase. In oil- and gas-dominated systems, hydrate interaction forces mainly originate from the

formation of water capillary bridges between hydrate particles; on the other hand, in water dominated systems, the hydrate interactions are the product of solid–solid cohesion. It has also been shown that an increase in contact time may induce the sintering of hydrate particles and thus significantly increase the cohesive forces (Aman et al. 2011; Aman et al. 2012a; Hu and Koh 2017). A schematic of a high-pressure MMF apparatus and its main components are depicted in Fig. 1.8a. Figure 1.8b shows the general trend of the variation of cohesive force as a function of contact time for a pair of gas hydrate particles in a hydrocarbon liquid phase at high pressure (Hu and Koh 2017).

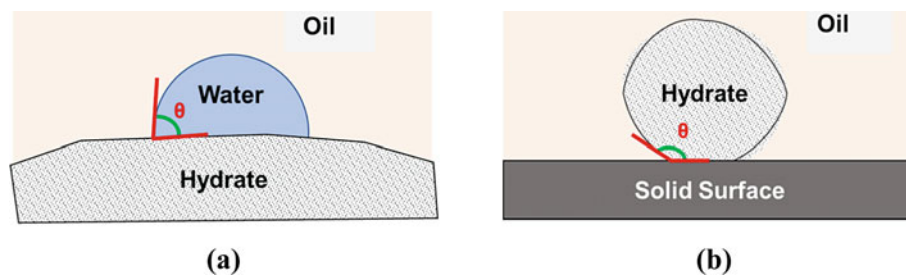
Measurements of cohesive forces have also been used to evaluate the effectiveness of natural and commercial AAs in oil-dominated systems, taking into account experimental parameters such as additive concentration, salinity, contact time and oil nature. Results indicate that the better the anti-agglomerant, the lower the interactions between hydrate particles (Aman et al. 2012b; Dieker et al. 2009; Hu and Koh 2020; Morrissy et al. 2017; Wang et al. 2020).

**Contact angle.** As depicted in Fig. 1.4, there are some techniques and setups applicable almost exclusively to systems with a significant amount of oil; the first of these is measuring the contact angle. Measurements of the contact angle ( $\theta$ ) of a water droplet placed on a hydrate surface/particle (see Fig. 1.9a) will give an indication of the wettability of the hydrate surface, thus a higher contact angles may be correlated to hydrophobic surfaces (Brown et al. 2018). This finding is very important considering that hydrate plugging in oil-dominated systems has been associated with the occurrence of water-wet hydrates at a low contact angle (Aspenes et al. 2010b). The contact angle technique has also been used to determine the hydrate-philicity of metal surfaces (Fig. 1.9b). According to the criterion mentioned above, higher contact angles account for low affinities between hydrates and solid surfaces (Brown et al. 2017). It has been



**Fig. 1.8** a Schematic of a high-pressure MMF apparatus (reproduced with license granted by the American Chemical Society from Hu and Koh 2017). b Illustration of typical variation of cohesive forces versus

contact time for gas hydrate particles in a hydrocarbon liquid phase at high pressure



**Fig. 1.9** Contact angle measurements **a** between a water droplet and a hydrate particle; **b** between a hydrate particle and a solid surface

recently demonstrated using contact angle measurements, among other experimental techniques, that the application of coatings and surface chemical treatments can efficiently reduce hydrate adhesion to metal surfaces (Brown et al. 2017; Das et al. 2019; Pickarts et al. 2019).

**Rocking cells.** A rocking cell apparatus is one of the most common devices used for flow assurance studies, and is another item used exclusively in oil-dominated systems. As the name suggests, it consists of a relatively small high-pressure visual cell coupled with a rocking mechanism. These are generally designed to operate at pressures of up to 5,000 psi and are often equipped with a metal ball to facilitate the mixing of phases; in some devices, the time required for the ball to travel from one end of the cell to the other end is recorded by two run-time sensors. Pressure and temperature may be recorded, which allows induction time, gas consumption, water conversion to hydrates and hydrate agglomeration to be determined (Chua and Kelland 2013; Dong et al. 2017; Frostman 2000; Sloan and Koh 2007). Rocking cells have been widely used to evaluate the performance of AAs as well as THIs; the visual assessment of hydrate agglomeration is based on the size and flowability of hydrate particles so that the results may vary from a “pass” to a “fail” test with one or more intermediate grades (Costa Salmin et al. 2019; Delgado-Linares et al. 2020; Gao 2008, 2009; Gupta et al. 2011). The effect of variables such as water cut, salinity, AA concentration and subcooling on hydrate agglomeration can be determined by using this kind of apparatus.

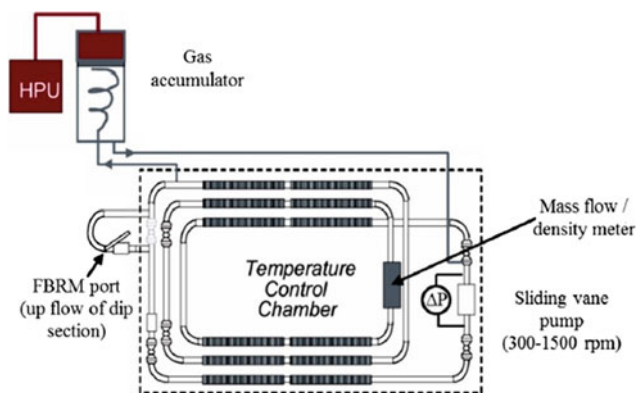
Some authors have proposed a variation of the rocking cell concept to study hydrates at different flowing conditions (e.g. a 1–5 L rock-flow cell larger than the conventional  $\sim 35$  mL rocking cell) with capabilities to modify the rocking angle/speed to provide different flow regimes (Sa et al. 2019).

**Bottle test.** The final technique covered here exclusive to oil-dominated systems is the bottle test, which is used to evaluate the stability of emulsions. In the classical bottle test, the volume of the phases when separated from an emulsion is recorded over time. For water-in-crude oil emulsions, the volume of the internal phase (water) that has separated is

registered, as well as other aspects related to the separation such as appearance of the interface and clarity of the separated water. Due to its simplicity, bottle tests enable field operators to obtain information about the kinetics of emulsion separation in a relatively short period of time. This technique has also been successfully used to evaluate the performance of commercial demulsifiers in the oil industry (Delgado-Linares et al. 2016; Goldszal and Bourrel 2000; Leopold 1992; Salager 1990; Smith and Arnold 1987; University of Texas 1990). Published works have suggested a relation between the stability of water-in-oil emulsions and gas hydrate transportability in crude oil systems; as a consequence, the bottle test is an important tool for potentially evaluating natural hydrate anti-agglomeration (i.e. naturally occurring surfactants responsible for the stabilization of crude oil emulsions may play a key role in gas hydrates dispersion) (Costa Salmin et al. 2019; Delgado-Linares et al. 2020; Lachance 2008; Salager and Forgiarini 2012; Sjöblom et al. 2010). The main limitation of this technique is that it is generally not performed at the high pressures of gas hydrate formation.

### 1.2.2.2 Pilot Scale

**Flow loop.** Industrial and research institutions have constructed pilot-scale flow loops to simulate the flow behavior in pipelines, and can thus obtain results closer to those in real-world conditions, as shown in Fig. 1.4. These apparatuses control the temperature and track hydrate formation kinetics by observing gas consumption at constant pressure and/or volume, pressure drops, and visually using windows/particle probes. Data acquisition systems record data from thermocouples, pressure sensors, flow meters, in-situ imaging probes, and windows for visual observation (Costa Salmin 2019; Sloan and Koh 2007). The total volume of flow loops is variable, but it may be in the range of 80–670 L, with a required volume of oil between 28 and 445 L (Costa Salmin 2019). Some of the flow loops used in hydrates studies worldwide include the ExxonMobil Friendswood flow loop in Texas (U.S.A) with 93 m length and 9.7 cm of internal diameter, the IFP flow loop in Solaize (France) with 140 m length and 5 cm of internal diameter, the University of Tulsa



**Fig. 1.10** Schematic of ExxonMobil flow loop with its main components (reproduced with license granted by Elsevier from Joshi et al. 2013)

flow loop in Tulsa (U.S.A.) with 49 m length and an internal diameter of 7.6 cm, and the flow loops at SINTEF Multiphase Flow Laboratory in Trondheim (Norway) with three loop facilities, namely one of small scale (50 m length with 2.54 and 5.08 cm of internal diameters), one of medium scale (50 m length with 6.35, 7.62 and 10.16 cm of internal diameters) and one of large scale (800 m length and 20.32 cm of internal diameter) (Anon 2020; Boxall 2009; Giavarini and Hester 2011; Sloan and Koh 2007). Figure 1.10 displays a schematic of the ExxonMobil flowloop and its main components (Joshi et al. 2013).

Flow loop tests have been carried out to evaluate the hydrate transportability for oil- and gas-dominated systems; the formation of agglomerates and deposits are detected by an increase in pressure drop (Di Lorenzo et al. 2014; Majid et al. 2016). Hence, flow loops can be a valuable tool to evaluate the effectiveness of hydrate AAs (Dapena et al. 2017; Lachance et al. 2012).

**Wheel loop.** An apparatus smaller than an industrial flow loop is a wheel loop. It is commonly comprised of a wheel/torus of 2–5 inch pipe at a diameter of 2 m, with a rotation velocity of 0.3–5 m/s when filled with gas and liquid (<50% liquid loading). Hydrate formation is determined visually or by a sharp increase in the torque required for rotation (Sloan and Koh 2007). The torque data may be used to evaluate the effect of AAs on the hydrate plugging potential of oil-dominated systems (Hemmingsen et al. 2008; Kelland et al. 2006).

### 1.3 Final Considerations

Gas hydrates have become a major topic of research for industry and academia during the last several decades, mainly due to their enormous potential as an energy resource and the necessity to avoid/manage them in oil and gas

flowlines. Much effort has been dedicated to understanding how hydrates form and behave in different systems where the dominant phase may be liquid or gas, and where the flow conditions are variable.

This multiscale approach has been developed by the Center for Hydrate Research (Colorado School of Mines) after many years of intensive research on gas hydrates in flow assurance. The vision outlined here will allow for the incorporation of the described phenomena/mechanisms studied at microscale into integrated models developed to explain and predict hydrate behavior in real-world conditions. It is, however, a large task given the high numbers of parameters to consider. The unification and scaling-up of microscopic models to robust predictive models applicable in field situations will be the key challenge for hydrate researchers in upcoming years.

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