

Experimental data and approximate estimation for dissociation time of hydrate plugs

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Abstract

New experimental data coupled with a numerical model and an approximate solution are proposed to predict dissociation time of hydrate plugs in oil sub-sea pipelines. The experimental hydrate plugs are dissociated by the method of symmetric depressurisation, both in a specially designed apparatus and a classical batch reactor. The agreement between the estimation of the model and the experimental data and the simplicity of the approximate equation presents an advantage in estimating the time of hydrate plug dissociation in pipelines.

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1. Introduction

Since the discovery of hydrates as a laboratory curiosity in 1811 by Sir Humphrey Davy, knowledge of hydrates has been slowly expanding continuously. Gas hydrates crystallise when natural gas contacts liquid water at high pressure and low temperature. These compounds are known for their ability to form blockages in hydrocarbon production systems. For instance, natural gas hydrates plugs cause problems during drilling, well operations, production, transportation and processing of oil and gas. Especially, it is a very serious problem in off-shore oil transportation where low temperature and high pressure become more and more favourable to gas hydrate formation as the new production wells are increasingly deeper.

Up to now, although many studies have been developed concerning thermodynamics and kinetics of gas hydrates crystallisation and on the possibility of preventing pipe plugging, there is limited information in open literature on hydrate plug's dissociation once they are formed.

In 1998, Kelkar et al. (1998) presented the first model of hydrate plugs dissociation, which was based on one-dimensional heat conduction in Cartesian coordinates. The mathematical

model developed in the work of Peters et al. (2000) was an extension of the previous modelling to a finite medium and cylindrical coordinates. Esben et al. (2002) proposed a Quasi Steady-State model to both Cartesian and cylindrical coordinates. He simplified the work by dividing the system into sub-systems that are assumed at steady state. All these models are numerical.

In our study, we develop firstly a numerical model to finite media and cylindrical coordinates. This model is based on the enthalpy method. Then, we propose an approximate equation. These two solutions are compared very well not only with our experimental results, which are presented here, but also with literature data.

2. Experimental devices and procedures

Materials. Ethane is from Air Liquide (France) and has a certified purity higher than 99.95 vol%. The *n*-dodecane is from V3-Chimie (France) and has a certified purity higher than 97 wt%.

Apparatus and experimental procedures. We have used two different apparatus systems to study the dissociation of hydrate plugs whose diameters are different.

The first one (A) is a classical batch reactor. The cell is a pyrex cylinder that is filled with a mixture of water and

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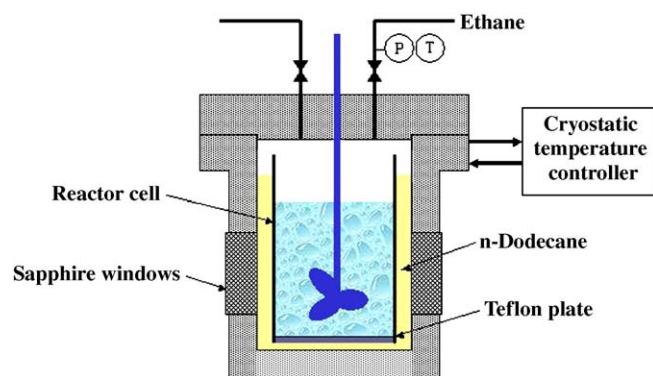


Fig. 1. Schematic of the batch reactor in the 1st experimental set-up (A).

n-dodecane. The cell has 0.2 m height, 0.003 m thickness and 0.1075 m diameter. It is located in a stainless steel autoclave in which the pressure can be raised up to 10 MPa. The autoclave is surrounded by a cooling jacket through which ethanol is circulated from a cryostatic temperature controller (temperature controlled within 0.02 K). A Teflon plate (0.01 m thick) covers the bottom of the cell in order to insulate hydrate plugs against heat source from the bottom. Two sapphire windows (0.1 m height, 0.015 m width) mounted on both sides of the reactor make possible observation of crystals and determination of hydrate plug height. A four vertical-blade turbine impeller ensures stirring of the suspension during crystallisation. The rotation rate can be varied from 0 to 600 rpm (Fig. 1).

The second experimental set-up (B) has been specially designed for this work. In system B, we used two reactors: the first reactor is a classical batch reactor and the second reactor named “main reactor” is described as follows. The measurement cell (462 cm³) of the main reactor is a transparent polycarbonate tube of internal diameter 0.07 m. It is located inside a tank of 101 that can be pressurised up to 28 MPa. It can operate in the temperature range of 253.15–423.15 K. The temperature control is ensured by circulation of an ethanol–water mixture in the volume separating the cell external surface (diameter 0.08 m) and the tank internal surface (diameter 0.15 m). Two Teflon plates are also mounted at the top and the bottom of the cell in order to thermally insulate hydrate plugs against heat source. Two pairs of windows (diameter 0.01 m) on both sides of the reactor allow us to see the cell. Because the cell is transparent, we can observe plug’s dissociation (Fig. 2).

Temperatures of the two set-up are monitored by Pt100 probes. The classical batch reactor (A) is monitored only with a Pt100 probe in the bulk. The set-up B is monitored with five Pt100 probes (Fig. 3).

Pressures of the set-up A are measured by means of pressure transducers (range: 0–10 MPa). The pressure uncertainty is estimated to be within $\pm 0.4\%$ of full scale (± 0.04 MPa). Pressures of the set-up B are measured by means of pressure transducers (range: 0–30 MPa). The pressure uncertainty is estimated to be within $\pm 0.25\%$ of full scale (± 0.075 MPa).

Temperature and pressure measurements are displayed or controlled by West instrument (model 8010 and 6100). The

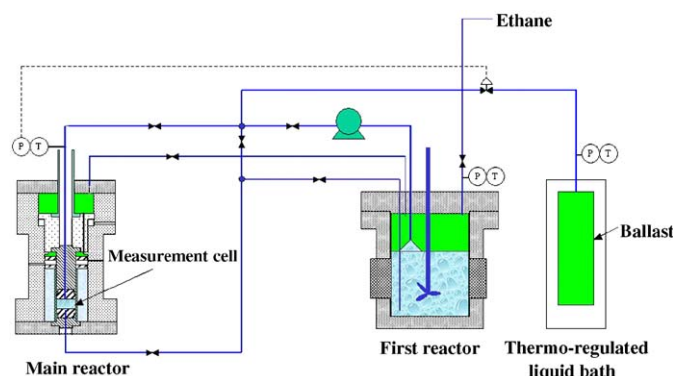


Fig. 2. Schematic of the 2nd experimental set-up (B).

data acquisition unit is also connected to a personal computer through two cards National Instruments (PCI-6023E and Lab1200).

In both set-ups A and B, the initial hydrate slurry is crystallised from 0.6 kg of water (66% wt) in *n*-dodecane mixture. In this study, *n*-dodecane is considered as oil and its presence makes the transferring of hydrate slurry easier. We obtain a homogeneous emulsion thanks to the addition of dispersant E102B furnished by IFP. The concentration of additive is 0.5–1.0% mass of the oil mass. In the beginning, the emulsion is cooled at atmospheric pressure, down to 277.15 K while stirring at 200–400 rpm. Then the pressure is increased up to a maximum value of 3 MPa and ethane supply is closed. We observe at first a temperature increase due to gas compression and then a pressure drop due to ethane dissolution. After that, the pressure decreases to reach the operative pressure and we observe a sudden temperature increase. This is the signal of appearance of the first hydrate crystals. Then the gas is consumed by the crystallisation, and the temperature slightly return to the operative condition (Fig. 4).

In the experimental set-up A, hydrate plugs are formed directly in the reactor. After complete crystallisation, stirring is stopped and hydrates accumulate at the bottom. In system B, the hydrates are formed in the first reactor and transferred to the measurement cell in the main reactor where they are accumulated to form a hydrate plug (Fig. 5).

The dissociation is operated as follows. The pressure is suddenly and manually decreased to the dissociation pressure (Point a, Fig. 6). When the pressure reaches the dissociation pressure, we open the valve between the reactor and a temperature-controlled ballast. The exhausting gas is transferred to the ballast whose pressure increases suddenly. Then the pressure of dissociation cell is maintained at a constant value by using an electrovalve and a pressure controller. The ballast is regularly emptied to maintain a sufficient pressure drop between the cell and the ballast (technical consideration due to the electrovalve which controls the pressure). By measuring pressure and temperature of the ballast (whose volume is known), we can determine the mass balances (Fig. 6). The measurement of the total quantity of gas released after dissociation coupled with the visual observation of the plug height

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