The Effect of the Hydrate Antiagglomerant on Hydrate Crystallization at the Oil–Water Interface

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ABSTRACT: Clathrate hydrates are ice-like compounds consisting of small gas molecules enclosed in water molecule cages. The formation of gas hydrate in oil and gas pipelines may result in flow assurance failure and serious safety and environmental concerns. Antiagglomeration is a promising method to mitigate gas hydrate risks in hydrocarbon flowlines. Morphological behavior of hydrates in the presence of antiagglomerants can provide important information on the antiagglomeration mechanisms. This study reports the visual observations of the morphology of hydrate formed with a water droplet immersed in cyclopentane with and without the presence of a hydrate antiagglomerant (AA). The effect of AA on the hydrate crystal growth was investigated. The AA exhibited a kinetic inhibition effect. With no AA, a faceted hydrate shell formed around the water droplet was observed. The subcooling can affect the rate of lateral growth. Higher subcooling facilitates hydrate growth. With the presence of 0.04 wt % AA, a hairy and porous morphology of hydrate was observed. At higher AA concentrations, a vertical type of growth after the lateral growth of the hydrate shell was observed. This is probably the first report of vertical growth of cyclopentane hydrate formed with a water droplet. A hypothesis is proposed to explain the vertical growth mechanism of the hydrate crystals.

INTRODUCTION

Clathrate hydrates are crystalline, ice-like compounds composed of small gas molecules enclosed in hydrogen-bonded water cages. The formation of gas hydrate in hydrocarbon flowlines often causes flow assurance failure, serious safety risks, and environmental concerns.1 Hydrate antiagglomeration by hydrate antiagglomerants (AAs) is a promising solution to mitigate gas hydrate risks in oil and gas pipelines. AAs affect the hydrate surface properties and prevent the aggregation of the small hydrate crystals into large, flowline-blocking masses and ensure that hydrate crystals are dispersed in the production stream. The AAs can exhibit higher performance at high subcooling conditions (e.g., >10 °C) than kinetic hydrate inhibitors.2 The main limitation associated with AA is that they are ineffective at high water cut conditions (e.g., >50%).3 New AAs that can be active at high water cut conditions remain to be developed.

In recent years, a novel AA proposed by Sun and Firoozabadi,4 where the effective component is cocamidopropyl dimethylamine, was confirmed to be effective at water cut up to 100% with respect to the liquid phase (with water to hydrate conversion less than 30 at 100% water cut).5−7 The high performance of this AA at high water cut conditions even in the bulk water phase is considered to be attributed to the strong binding force between the AA molecule and hydrate surface. Based on the investigations conducted by the research groups of Firoozabadi1−9 and Chen et al.,10 AA was confirmed to be effective in hydrocarbon/gas (methane or natural gas)/aqueous mixtures at high pressures up to 10 MPa and water contents up to 100% (with respect to the liquid phase). However, emulsions stabilized by AA were observed to be not stable. An emulsion (water-in-oil) with high stability produced by AA can facilitate the hydrate antiagglomeration by predispersing the continuous water phase into suspended water droplets in the oil phase. The low stability of the emulsion produced by AA may demonstrate the low density of adsorbed AA molecules at the water−hydrocarbon interface.11 As described above, the AA’s performance in a sapphire rocking cell setup at high pressure conditions has been extensively rated. However, the rocking cell test is considered to be time-consuming and may not provide all the detailed results that we need to understand the performance of AA. The effect of AA on hydrate formation at the water−oil interface, which plays a critical role in hydrate antiagglomeration, remains to be further investigated. Additionally, it was demonstrated that the visual observations of the morphological behavior of hydrates in the presence of antiagglomerants can provide important information on the antiagglomeration mechanisms.12 The morphology of gas hydrate crystals can be significantly changed by AAs.12 The formation of hydrate is considered to be an interfacial phenomenon and occurs at the interface between water and the hydrate former phase. A three-
step mechanism (nucleation, lateral growth, and radial growth) has been proposed for the hydrate formation and verified by many researchers. These considerations motivated us to obtain further insight into the working mechanisms of AA by conducting visual observations of the nucleation and growth of hydrate crystals on the surface of a water droplet exposed to the oil phase with and without the presence of AA.

In this study, cyclopentane is employed as the hydrate former as it is immiscible in the water at atmospheric pressure and forms structure II hydrate (the same hydrate structure as that encountered in flowlines). A self-hydrate-visualization cell apparatus is used to study the influence of AA on the morphology of cyclopentane hydrate. The growth mechanism of cyclopentane hydrate in the presence of AA is also proposed.

**EXPERIMENTAL SECTION**

**Chemicals.** Cyclopentane (CyC5) serving as the hydrate former was purchased from Aladdin with a purity of 96 wt %. The cyclopentane hydrate is stable below 7.7 °C, which can ensure that the experiments can be conducted above the ice point to avoid the ice formation during the tests. The hydrate antiagglomerant (AA) used in this study was obtained from Lubrizol Specialty Chemicals (Shanghai) Co., Ltd. It contains 80–89 wt % cocamidopropyl dimethylamine (see Figure 1).

![Chemical structure of the effective component (cocamidopropyl dimethylamine) of AA](Image 1)

Figure 1. Chemical structure of the effective component (cocamidopropyl dimethylamine) of AA (n = 10−12).

5–10 wt % glycerin, a small amount of free amine, and water. The AA was used as received without further purification. DI water was obtained from a laboratory purification system (Continental Water System).

**Hydrate-Visualization Cell Apparatus.** In the present study, the nucleation and growth of the cyclopentane hydrate were artificially induced in a hydrate-visualization cell. Figure 2 schematically illustrates the hydrate formation and recording system. An X90 pipeline steel plate (1 cm × 1 cm × 0.2 cm) polished using 2000 grit sandpaper is placed in a transparent rectangular quartz cell. The compositions of the X90 steel plate are listed in Table 1. The quartz cell is equipped with a cooling jacket and filled with cyclopentane or cyclopentane solution with AA. An external chiller (DC-2006, Ningbo Scientz Biotechnology) with a precision of ±0.1 °C is connected to the cooling jacket to control the temperature of the system. At the beginning of each test, the cyclopentane bath is maintained at −3 °C. A distilled water droplet (3 μL) is placed on the steel plate, and the cyclopentane bath is then heated to 1 °C (or 4 °C) at 1 °C/20 min and kept at constant temperature conditions for 30 min. A small portion of preformed hydrate seed crystals (5 mg) is then placed at the cyclopentane−water interface with a 50 μm diameter steel wire to initial hydrate nucleation. The cyclopentane is kept at 1 °C (or 4 °C) for a certain length of time, allowing the hydrate to fully develop. In each test, the hydrate formation occurs under static conditions, and the effects of heat transfer are considered to be ignorable. During the hydrate formation and growth, a microscope with a CCD camera (DV-100) is used to record a video of the morphology and growth behavior of the hydrate crystal. Prior to each test, the cell and the steel plate should be rinsed with distilled water and acetone and finally dried using a heat gun.

| Table 1. Element Composition of X90 Pipeline Steel (wt %) |
|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| C           | Si          | Mn          | Cr           | Mo          | Ni          | Cu          | V + Nb + Ti |
| 0.05        | 0.40        | 1.60        | 0.30         | 0.30        | 0.30        | 0.30        | 0.10        |

**RESULTS AND DISCUSSION**

**Surfactant-Free Case.** Figure 3 shows the sequence of the growth of cyclopentane hydrate crystals from a distilled water droplet immersed in the cyclopentane bath without AA at 1 °C. The water contact angle on the polished X90 pipeline steel plate is determined to be 94.1° (Figure 3a) through ImageJ. Shortly after the introduction of a hydrate crystal seed on the top of the water droplet, the hydrate nuclei grow laterally along the cyclopentane−water interface, leading to a polycrystalline hydrate shell as shown in Figure 3c. The hydrate shell spreads from the top of the water droplet and finally encapsulates the water droplet. Due to the high subcooling (6.7 °C), the water droplet was fully covered by the hydrate shell within 10 min after the hydrate crystal seeding. On the X90 pipeline steel surface, around the water droplet, a thin hydrate film formed outward from the three-phase contact line is observed (Figure 3g). The thin hydrate film may be caused by the release of unconverted water from the interior of the hydrate shell. A similar phenomenon was observed by Farnham et al.22

Figure 4 shows the sequence of hydrate formation and growth of cyclopentane hydrate from a water droplet similar to those in Figure 3 at a higher temperature (4 °C). After the seed hydrate crystals were placed on the cyclopentane−water interface (Figure 4a), no rapid hydrate growth was observed. In the test, the seed hydrate crystal was observed to gradually move to the bottom of the water droplet (to the three-phase contact line); during this process, no noticeable hydrate growth induced by the seed crystal was observed, which may be due to the low subcooling. As can be seen from Figure 4bc, both the steel and the seed crystals can induce the hydrate formation.
growth. The hydrate that starts to form at the steel surface may be nucleated by the seed hydrates that stick to the steel surface. As recognized by Figures 3 and 4, the time required for a full encapsulation of the water droplet with the hydrate shell depends on the subcooling. Due to the low subcooling ($3.7^\circ C$), which acts as the driving force to propagate the hydrate front at the cyclopentane−water interface, the encapsulation of the water droplet by the hydrate shell was completed in 1 h (Figure 4e), while only 10 min was needed at $1^\circ C$ as shown in Figure 3c. As shown in Figure 4h, after the complete coverage, no further growth of the hydrate shell was observed in 4 h.

It is also evident that the subcooling affects the morphology of the hydrate crystals covering the interface. In Figures 3 and 4, hydrate crystals grew along the interface and formed a polycrystalline shell covering the water−cyclopentane interface. At $1^\circ C$, for $\Delta T_{sub} = 6.7^\circ C$, a hydrate shell with a rough surface was observed (Figure 3), while at $4^\circ C (\Delta T_{sub} = 3.7^\circ C)$, a faceted hydrate shell was observed. In Figure 4, the hydrate shell was smooth compared to that observed in Figure 3, which may be due to the bigger size of the facets in the hydrate shell. Kishimoto et al.24 reported that the facet size of the faceted cyclopentane hydrate layer formed at the interface between water and liquid cyclopentane decreased with subcooling.

**Effect of the Hydrate Antiagglomerant (AA) at 1 °C.**

The effect of AA on the cyclopentane hydrate morphology is investigated in this section. In Figure 5, the AA concentration and temperature are fixed at 0.04 wt % (based on the weight of the cyclopentane phase) and $1^\circ C$, respectively. In Figure 5,
the entire coverage of the water−cyclopentane interface by hydrate crystals occurs at ∼30 min after seeding, indicating that AA could retard the growth of the hydrate crystals. A hairy and porous hydrate morphology was observed as compared with the faceted shell formed at the water−cyclopentane interface in the absence of AA (Figure 4). A similar hairy hydrate morphology caused by the surfactant was also observed by Karanjkar et al., Nakajima et al., and Aman et al. Aman et al. observed a hairy morphology of tetrahydrofuran hydrate in the presence of Span 20 and hypothesized that the changes in the hydrate morphology may be attributed to the presence of surfactants in the bulk liquid phase rather than the hydrate former. Here, we assume that the formation of a hairy and porous morphology is caused by the presence of insufficient AA. The AA molecules are considered to adsorb onto the water−cyclopentane interface before the seeding of the hydrate crystals at the interface due to its amphiphilic nature. Once the cyclopentane hydrate appears at the interface, some of the AA molecules migrate from the water−cyclopentane interface to the hydrate surface due to its hydrate-philic nature. After the complete coverage of the interface with hydrate crystals, vertical growth of the hydrate crystals was observed as shown in Figures 6 and 7. In the past, the nucleation, lateral growth, and radial growth have been widely reported by many researchers. To the best of our knowledge, such an interesting structure has not been reported before.

We further increased the concentration of AA in the cyclopentane bath. Figures 6 and 7 show the sequential images similar to those in Figure 5 at higher AA concentrations at 1 °C: 0.08 wt % for Figure 6 and 0.12 wt % for Figure 7. In Figures 6 and 7, after the seeding of the hydrate crystals, the entire coverages of the water−cyclopentane interface with the hydrate crystal occur at ∼45 and ∼60 min after seeding, respectively. These observations indicate that AA could further retard the hydrate growth. It should be noted that, due to the presence of the AA in the cyclopentane bath, the lateral growth of the hydrate crystals was delayed. Without AA, the lateral growth occurs shortly after the seeding of the hydrate crystals as shown in Figure 3. With the presence of 0.08 wt % AA in the cyclopentane bath, the noticeable lateral growth of hydrate crystals starts in 2 min after the seeding of the hydrate crystals (Figure 6). After 3 min of seeding, the noticeable lateral growth of the hydrate crystals starts in the presence of 0.12 wt % AA (Figure 7).

As recognized in a comparison of Figures 5–7, the hydrate crystals display an interesting structure when the AA concentration is higher than 0.04 wt %. After the complete coverage of the water−cyclopentane interface by the hydrate shell (lateral growth), vertical growth of the hydrate crystals was observed as shown in Figures 6 and 7. In the past, the nucleation, lateral growth, and radial growth have been widely reported by many researchers. To the best of our

Figure 6. Sequential images of hydrate shell growth at the interface between water and cyclopentane in the presence of 0.08 wt % AA, T = 1 °C: (a) 0 min; (b) 5 min; (c) 10 min; (d) 30 min; (e) 1 h; (f) 2 h; (g) 4 h; (h) 6 h.

Figure 7. Sequential images of hydrate shell growth at the interface between water and cyclopentane in the presence of 0.12 wt % AA, T = 1 °C: (a) 0 min; (b) 5 min; (c) 10 min; (d) 1 h; (e) 2 h; (f) 4 h; (g) 8 h; (h) 16 h.
knowledge, this is probably the first report of vertical growth of the cyclopentane hydrate crystals in the presence of a special surfactant (AA). The vertical growth of the hydrate crystals may be caused by subcooling and the strong adsorption of AA molecules on the surface of the hydrate shell and X90 pipeline steel, which will be explained in detail shortly.

**Effect of the Hydrate Antiagglomerant (AA) at 4 °C.**

Figure 8 shows the sequential images similar to those in Figures 6 and 7 at a higher temperature: 4 °C ($\Delta T_{sub} = 3.7$ °C). In Figure 8, 2 h after the seeding of the hydrate crystals, the noticeable lateral growth along the water–cyclopentane interface was observed as shown in Figure 2c. Before the lateral growth starts, the water droplet gradually spreads on the X90 steel surface, forming a thin water layer. The formation of the water layer may contribute to the decrease in water–cyclopentane interfacial tension, which could soften the interface. The coverage of the water–cyclopentane interface with the hydrate crystal was complete within 16 h after the introduction of the hydrate seeds. These observations indicate that the AA product also exhibits a kinetic inhibition effect. It should be noted that the upward growth of the hydrate crystals was observed. After the complete water conversion, the hydrate crystals display a mountain-like morphology due to the vertical growth of the hydrate. The formation of mountain-like crystals will be explained shortly.

**Proposed Vertical Growth Mechanism of the Hydrate Crystals.** The formation of a gas hydrate particle formed with a water droplet is considered to involve nucleation, lateral growth, and radial growth.13−20 In addition to these steps, vertical growth of the gas hydrate was observed in this study. A proposed vertical growth mechanism of the hydrate crystals is sketched in Figure 9 and described below.

Before the seeding of hydrate crystals on the water droplet immersed in the AA/cyclopentane solution, the AA molecules are mostly in the form of an adsorption layer covering the water droplet and the X90 steel plate as shown in Figure 9a. The adsorption of AA molecules onto the water–cyclopentane is due to its amphiphilic nature, while the adsorption of AA molecules onto the X90 steel surface is due to the chemisorption.27,28 Due to the presence of sufficient AA in the cyclopentane bath, some of the AA molecules are in the form of micelles. After the hydrate crystals are seeded on the water droplet and the lateral growth of the hydrate crystals starts, the residual water is encapsulated by the hydrate shell. Once the hydrate appears at the interface, AA molecules simultaneously adsorb onto the hydrate surface due to its hydrate-philic nature5,6 and form a closely packed adsorption layer after the formation of the hydrate shell (Figure 9b). After the lateral growth, the radial growth starts and leads to an increase in the pressure of the residual water inside the hydrate shell. The hydrate shell detaches from the steel surface because of the increased pressure of the residual water core, and hence, a new interface for hydrate formation is generated at the three-phase contact line. The newly formed interface locates at the bottom of the hydrate shell and is governed by the hydrate shell as water conversion occurs at the interface, and then radial growth restarts again, which would increase the pressure of the residual water inside the hydrate shell. The water–cyclopentane interface at the bottom of the hydrate shell is regenerated for further hydrate formation. The formation of the new water–cyclopentane interface repeatedly occurs until the complete conversion of the residual water to hydrate, and thus, the vertical growth of the hydrate shell is observed (Figure 9c).

The morphological changes of the hydrate shell caused by AA may give insight into the AA’s working mechanism. In Brown’s thesis,12 the least effective AA was found to produce
almost no visible changes in the morphology of cyclopentane hydrate, while the highly effective AAs would lead to a significant change in the hydrate morphology. Based on the morphological changes of hydrate in the presence of AAs, a working mechanism of the AA, which was different from the mechanism obtained in rocking cell tests, has been proposed. In this study, we found that the AA product can significantly affect the morphology of the hydrate shell, which may imply that the AA product is a highly effective hydrate antiagglomerant. The AA’s high effectiveness in hydrate antiagglomeration has been verified in some previous reports. These results from the observation of the morphological changes may provide a simple prediction of the AA’s performance.

### CONCLUSIONS

We conducted a set of observations of the morphology of hydrate formed with a water droplet immersed in cyclopentane with and without the presence of a hydrate antiagglomerant (AA). The effect of AA on the hydrate crystal growth was investigated. The AA exhibited a kinetic inhibition effect. With no AA, a faceted hydrate shell formed around the water droplet was observed. The subcooling can affect the rate of lateral growth. Higher subcooling facilitates hydrate growth. With the presence of 0.04 wt % AA, a hairy and porous morphology of hydrate was observed. At higher AA concentrations, a vertical type of growth after the lateral growth of the hydrate was observed. This is probably the first report on vertical growth of cyclopentane hydrate formed with a water droplet. A hypothesis is proposed to explain the vertical growth mechanism of the hydrate crystals.

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**Notes**

The authors declare no competing financial interest.

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