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Testing of Gravitational Stability of CO_2-Hydrates for CO_2 Storage on Seafloors

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Abstract

Discharging carbon dioxide (CO_2) to seafloors in the form of CO_2-hydrates can potentially store CO_2 in virtually unlimited quantity. Realizing the process requires a thorough understanding of CO_2-hydrate forming behavior in seawater conditions. One of the major concerns is the gravitational stability of the produced bulk CO_2-hydrates. The objective of this study was to seek the optimum procedure for producing bulk CO_2-hydrates with densities greater than seawater density. This objective was achieved by direct visualization of CO_2-hydrates through the glass windows of a reactor under various pressure and temperature conditions with mixing methods of CO_2-bubbling and water-dropping. CO_2-bubbling into the water phase was observed to produce bulk CO_2-hydrates with densities less than water density due to the excessive CO_2 trapped in the bulk hydrates. Water-dropping into the CO_2 phase was observed to produce bulk CO_2-hydrates with densities greater than water density due to the excessive water trapped in the bulk hydrates. Water-dropping was therefore identified as a favorable mixing method for producing CO_2-hydrates with gravitational stability for depositing onto seafloors.

Keywords

CO_2-Hydrates, Gravitational Stability, Experimental Study, CO_2 Storage, Seafloors

1. Introduction

Carbon Capture, Utilization, and Storage (CCUS) processes have gained significant momentum in recent years for reducing carbon dioxide (CO_2) content in the Earth's atmosphere. Carbon storage is realized by injecting CO_2 into geological structures, mainly depleted oil reservoirs [1]. This process takes advantage of using the pre-existing wells in Enhanced Oil Recovery (EOR) projects to inject CO_2

for cost reduction. Another advantage of using depleted oil reservoirs to store CO_2 is the high injectivity of CO_2 into oil reservoirs. A critical concern of using EOR wells as CO_2 receivers is the risk of CO_2 leakage through EOR wellbores due to the attack of CO_2 on the well cement sheath [2] [3]. The leakage is promoted by the high mobility (low viscosity) of supercritical CO_2 in high-temperature conditions that prevail in EOR wells. It has been practiced to inject CO_2 into sub-sea water zones where CO_2 should exist in liquid form in the low-temperature environment. This should significantly increase CO_2 viscosity and reduce the risk of CO_2 leakage [4]. Field-scale CO_2 injection projects include the Sleipnir project in the Norwegian Sea [5], the Hokkaido project in Japan [6] [7] and the Gulf Coast project in the United States [8]. Simulation studies have shown that the possibility of CO_2 leakage from the sub-sea water zones is very low [9]. However, CO_2 injection into the sub-sea water zones has a common issue of low injectivity due to the low-permeability nature of the water zones. To further reduce the risk of CO_2 leakage from its storage reservoirs, CO_2 storage in its hydrate form has been considered.

Small-molecule gases (e.g., methane, ethane, propane, iso-butane, CO_2, and H_2S) are trapped inside the cages of water molecules, forming gas hydrates under high-pressure and low-temperature conditions. The molecular size of these hydrophobic gases and volatile organic compounds is 3.8 Å to 9.2 Å. In appearance, the hydrates resemble snow or loose ice [10].

Injecting CO_2 into permeability zones where the in-situ temperatures are lower than the temperature required for hydrate formation allows for CO_2 storage in hydrate form, eliminating the possibility of CO_2 leaking through wellbores. This idea has been investigated in previous studies [11] [12]. However, it takes a lengthy time for CO_2 molecules to fully contact water molecules to form CO_2 hydrates due to the limited rate of mass transfer. In fact, this has been done in methane hydrate reservoirs for more than a decade in the CH_4-CO_2 swapping processes. The unsolved issue is that the CO_2 swapping rate is limited by the heat-transfer efficiency. Using geothermal energy to improve heat transfer efficiency may be a viable solution [13]. However, this idea is still in the stage of theoretical investigations.

The first common disadvantage of CO_2 carbon storage in geological structures, either depleted oil reservoirs or low-temperature water zones, is that wellbores are required for CO_2 injection, which increases the cost of operations. The second common disadvantage is the low capacity of storage due to the limited reservoir pore volumes. These issues have brought back the idea of depositing CO_2 directly onto sea/ocean floors [14]. If CO_2 is mixed with seawater near the sea floor in conditions that are favorable for hydrate formation, CO_2-hydrates should form and theoretically sink to the bottom of the sea/ocean by virtue of the relatively high density of the hydrate.

Guo *et al.* [15] presented an analytical method for determining the minimum seawater depth required to dispose of CO_2 in hydrate form on the seafloor. Amponsah [16] determined the minimum CO_2-storage seawater depths in the major

seas around the world and the Arctic Ocean. His work shows that the minimum required seawater depth varies greatly from 200 m to 650 m. The Persian Gulf is too shallow for CO_2-storage in hydrate form due to its high temperature. The minimum CO_2-storage seawater depth in the Arctic Ocean is about 120 m.

The CO_2 storage in hydrate form on the seafloor requires that the density of hydrates be greater than that of seawater. It is known that 1 m^3 of pure hydrates contains approximately 170 sm^3 of CO_2 depending on the formation process. This makes CO_2-hydrates heavier than seawater. However, free CO_2 (bubbles) can be trapped inside the bulk hydrates due to the excessive proportion of CO_2 during the chemical reaction in non-equilibrium conditions, making the density of bulk hydrates less than that of seawater [17]. Guo and Mahmood [18] proposed a jet-cooling method to accelerate the formation of CO_2-hydrates. Although the jet-cooling technique can generate CO_2 hydrates quickly, the generated bulk hydrates may trap CO_2 bubbles, making the bulk hydrates float and unable to settle down to the seafloor. On the other hand, free water may be trapped inside the bulk hydrates due to the excessive proportion of water during the chemical reaction in non-equilibrium conditions, making the density of bulk hydrates greater than that of seawater. Therefore, it is necessary to study the hydrate-forming processes to find a reliable and reproducible method to produce heavier-than-water CO_2 hydrates for CO_2 deposition onto seafloors.

2. Investigation Method

CO_2-hydrate formation under various conditions was observed through a windowed reactor shown in **Figure 1.** The reactor has see-through windows made of sapphire glass at both ends. The glass has a design strength of 400 MPa at 20˚C and 275 MPa at 500˚C, with a Young's modulus of 345 GPa and Poisson's ratio of 0.27. Its flexural strength (modulus of rupture) is 0.9 GPa to 1.9 GPa, with a Knoop hardness of 1525 - 2000. The reactor was designed to handle a stress (safe working stress) up to 150 MPa (22,000 psi) with a safety margin. The reactor's 3-inch diameter, 0.29 inch (7.4 mm) thick, sapphire glass plate can hold up to 2000 psi internal pressure. The reactor was water-pressure-tested up to 2000 psi for 24 hours with no leaks identified.

The whole observation system is illustrated in **Figure 2** with the windowed reactor drawn at the center. The ISCO metering pump provides water to the reactor through ports either at the top or bottom. The CO_2 bottle feeds the Accumulator with CO_2 gas up to 1000 psi. The ISCO metering pump squeezes water to the Accumulator so that CO_2 is displaced through a CO_2 Condenser into the reactor through ports either at the top or bottom. The Cooler with Pump circulates coolant through the coils around the reactor covered by an insulation layer. The back-pressure regulator controls the pressure in the reactor. Pressure and temperature inside the reactor are measured by the pressure gauge (PG), pressure transducer (PT), and temperature transducer (TT). These parameter values are stored and analyzed in the Computer.

Figure 1. Image of a reactor with see-through windows at both ends.

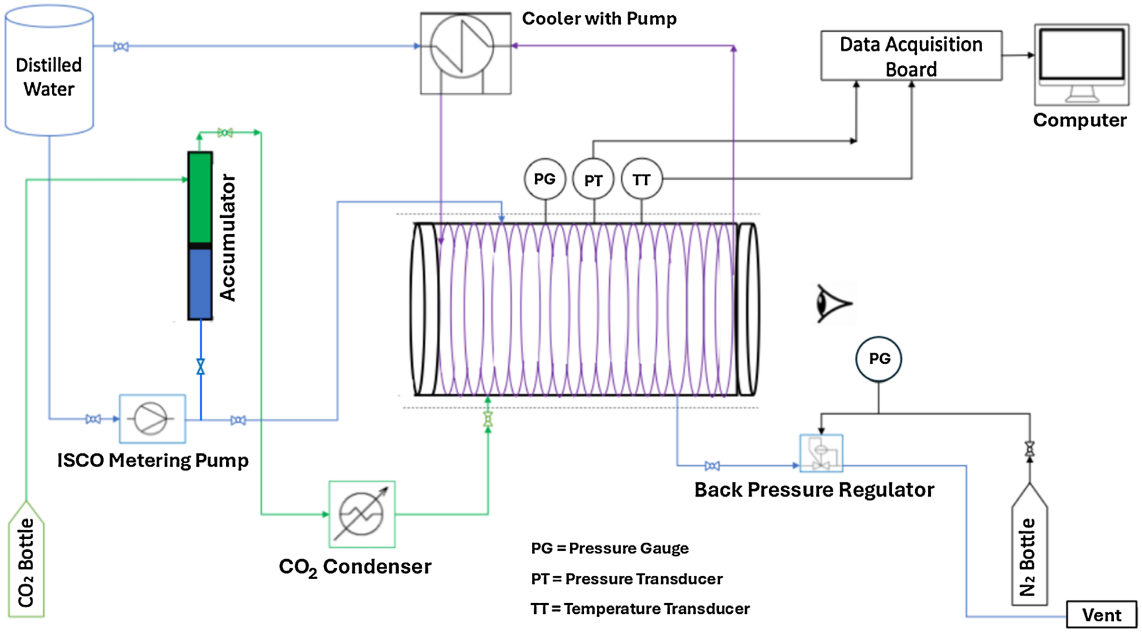


Figure 2. Schematic of the observation system.

CO_2 hydrate formation was observed in two mixing procedures: 1) CO_2-bubbling into the water phase and 2) water-dropping into the CO_2 phase. The procedure for CO_2-bubbling into the water phase is outlined as follows:

1) Fill the reactor with water and pressurize it to the desired pressure level using the ISCO metering pump.

2) Fill the upper section of the Accumulator with CO_2 using the CO_2 bottle.

3) Lower the reactor temperature using the Cooler with Pump until it reaches the desired temperature level.

4) Use the ISCO Metering Pumper to displace the CO_2 in the Accumulator through the CO_2 Condenser into the reactor through a 1/16 bottom port at 5 ml/min.

5) Observe CO_2-hydrate formation at different CO_2 injection rates through the reactor windows.

The procedure for water-dropping into the CO_2 phase is outlined as follows:

1) Fill the upper section of the Accumulator with CO_2 using the CO_2 bottle.

2) Use the ISCO Metering Pumper to displace the CO_2 in the Accumulator through the CO_2 Condenser into the reactor through a bottom port until it reaches the desired pressure level.

3) Lower the reactor temperature using the Cooler with Pump until it reaches the desired temperature level.

4) Inject water at a 5 ml/min flow rate into the reactor through a 1/16 top port using the ISCO metering pump.

5) Observe CO_2-hydrate formation at different water injection rates through the reactor windows.

3. Investigation Condition

Amponsah [16] determined the minimum water depths required for CO_2-storage in hydrates in 6 seas around the world and the Arctic Ocean. His results show that the minimum required water depth varies from 120 m in the Arctic Ocean to 650 m in the Mediterranean Sea. The temperatures at the minimum required water depth range from 0˚C in the Arctic Ocean to 11˚C in the Gulf of Mexico.

Figure 3 reproduces the static conditions for forming CO_2 hydrates [18]. It shows that, in the temperature range between 273.15 K (0˚C) and 284.15 K (11˚C) and the pressure range between 3.3 MPa (485 psi) and 5.4 MPa (800 psi), pure CO_2 exists in liquid form and liquid CO_2 can form hydrates when water is present. CO_2 hydrate formation was investigated in the temperature range from 0˚C to 3˚C and in the pressure range from 500 psi to 800 psi. Therefore, the observed CO_2 in this study is CO_2 liquid.

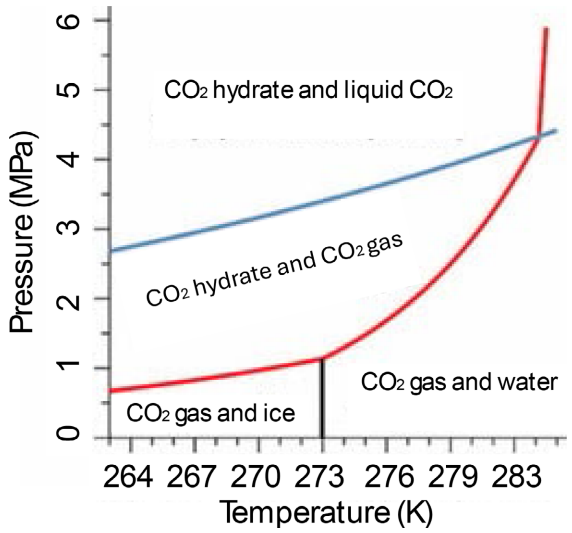


Figure 3. Static conditions for forming CO_2 hydrates [18].

4. Results

Figure 4 shows two images of CO_2 hydrate formation during CO_2-bubbling at 0.4˚C and 800 psi. The left image was captured when CO_2 entered the reactor in the form of bubbles at the bottom of the water phase. The right image was cap-

tured when hydrates formed around the CO_2 bubbles before the hydrate-shelled bubbles floated up to the top of the water phase. The CO_2 bubbles with hydrate-shells accumulated on the top side of the water phase, indicating that the bulk hydrates (pack of hydrate-shelled CO_2 bubbles) have a bulk density less than the water density. This is because the bulk hydrates contain liquid CO_2 as an excess phase.

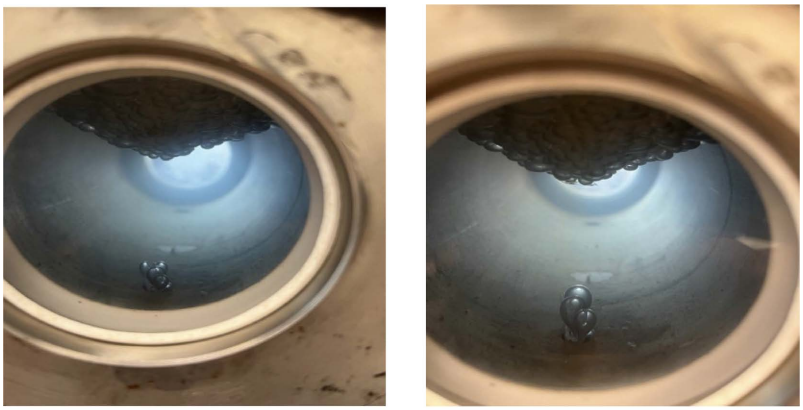


Figure 4. Images of CO_2 hydrate formation in CO_2-bubbling at 0.4˚C and 800 psi: (a) CO_2 enters the reactor in the form of bubbles, (b) hydrates form around the CO_2 bubbles before floating up.

Figure 5 shows two images of CO_2 hydrate formation during CO_2-bubbling at 2˚C and 800 psi. The left image was captured when a CO_2 bubble rose in the water phase. The right image demonstrates accumulations of CO_2 bubbles and hydrate-shelled CO_2 bubbles at the top of the water phase. The accumulation of hydrate-shelled CO_2 bubbles on the top side of the water phase implies that the bulk hydrates (a pack of hydrate-shelled CO_2 bubbles) have a bulk density less than the water density. Again, this is because the bulk hydrates contain liquid CO_2 as an excess phase.

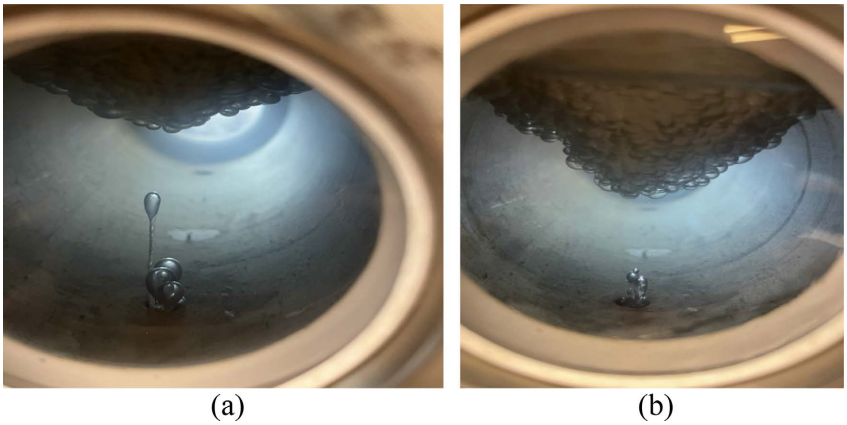


Figure 5. Images of CO_2 hydrate formation in CO_2-bubbling at 2˚C and 800 psi: (a) a CO_2 bubble floating up in the water phase, (b) accumulation of CO_2 bubbles and hydrate-shelled CO_2 bubbles.

Figure 6 presents two images of CO_2 hydrate decomposition when the reactor pressure was reduced from 800 psi to 500 psi at 2˚C (the equilibrium pressure is 515 psi). The left image shows the collapse of hydrate-shelled CO_2 bubbles. The right image shows collapsed hydrate shells floating at the interface between liquid CO_2 and water phases, suggesting that the residual hydrates still trapped liquid CO_2.

Figure 6. Images of CO_2 hydrate decomposition with a pressure reduction from 800 psi to 500 psi at 2˚C: (a) collapse of hydrate-shelled CO_2 bubbles, (b) collapsed hydrate shells.

Figure 7 presents two images of CO_2 hydrate formation during continuous CO_2 injection at 2˚C and 800 psi. The image on the left shows a hydrate-coated CO_2 channel at a 5 mL/min CO_2 flow rate. The image on the right shows a hydrate-coated CO_2 channel at a 10 mL/min CO_2 flow rate. It is seen that a straighter CO_2 channel formed at a higher CO_2 flow rate.

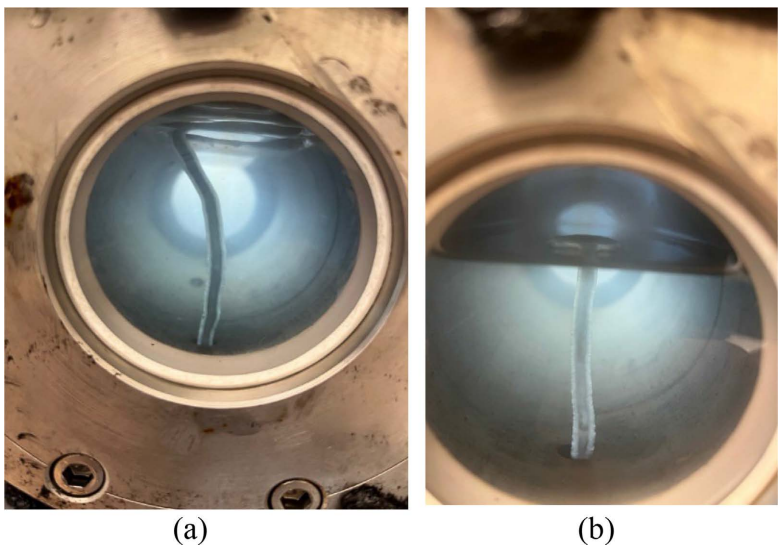


Figure 7. Images of CO_2 hydrate formation during CO_2-injection at 2˚C and 800 psi: (a) hydrate-coated CO_2 channel at 5 mL/min CO_2 flow rate, (b) hydrate-coated CO_2 channel at 10 mL/min CO_2 flow rate.

Figure 8 shows two images of CO_2 hydrate formation during water dropping at 0˚C and 500 psi. The image on the left indicates that a clear water pocket remained at the bottom initially. The image on the right shows CO_2-hydrate crystals at the bottom of the reactor after 1 hour of growth, indicating that the hydrate crystals have a density greater than that of water.

(a) (b)

Figure 8. Images of CO_2 hydrate formation during water dropping at 0˚C and 500 psi: (a) the water pocket stayed at the bottom of the CO_2 liquid at the beginning, (b) CO_2-hydrate crystals at the bottom side of the reactor after 1 hour.

Figure 9 shows two images of CO_2 hydrate formation after water dropping at 0˚C and 500 psi. The image on the left indicates CO_2-hydrate crystals grown from the water pocket after 4 hours. The image on the right shows CO_2-hydrate crystals everywhere in the reactor after 24 hours.

(a) (b)

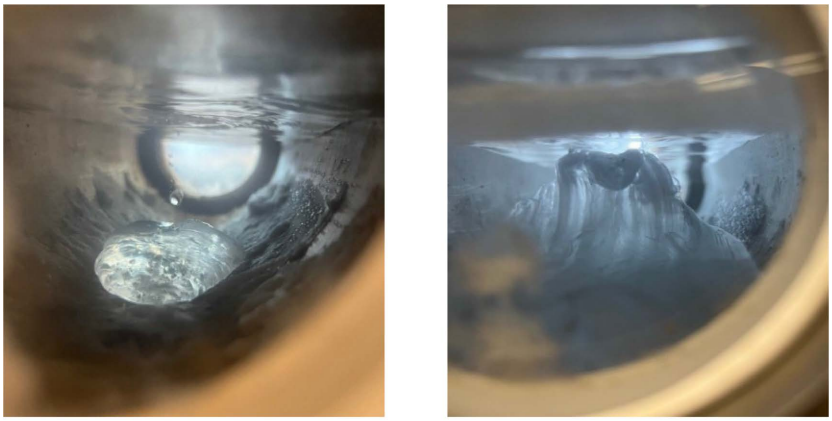
Figure 9. Images of CO_2 hydrate formation from water dropping at 0˚C and 500 psi: (a) after 4 hours, (b) after 24 hours.

Figure 10 presents two images of CO_2 hydrate formation from water dropping at 2˚C and 500 psi. The image on the left indicates the initial water pocket at the bottom. The image on the right shows CO_2-hydrate crystals remaining at the bottom after 3.45 hours.

Figure 11 shows two images of CO_2 hydrate formation from water dropping at 3˚C and 500 psi. The image on the left indicates the initial water pocket at the

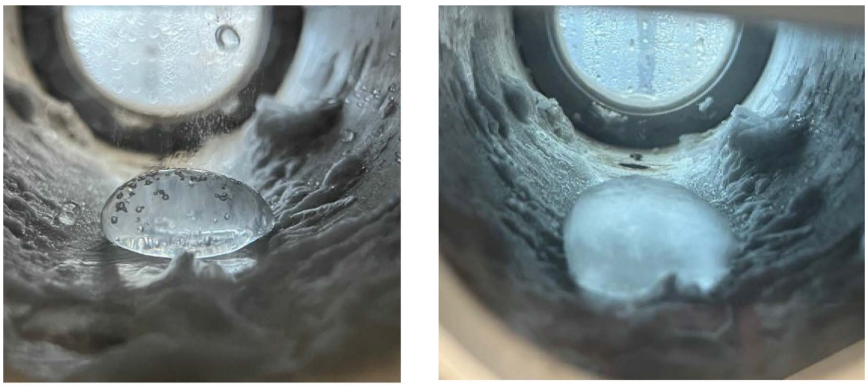
bottom. The image on the right shows a hydrate-shelled water pocket staying at the bottom after 12 hours. The slow formation of CO_2-hydrate is because this condition is very close to the equilibrium condition indicated by Figure 3. However, as shown in Figure 12, a large amount of hydrate crystals was found after 34 hours.

In summary, although the density of pure CO_2-hydrate is theoretically greater than that of water, the observed floating behavior of the bulk hydrates formed during CO_2-bubbling suggests that the density of the bulk hydrates is less than that of water. This is because free CO_2 is trapped inside the bulk hydrates and outside the hydrate crystal structure. On the other hand, the observed settling behavior of the bulk hydrates formed during water-dropping suggests that the density of the bulk hydrates is greater than that of water. This is because no free CO_2 is trapped inside the bulk hydrates and outside the hydrate crystal structure. Either some free water or no water is trapped inside the bulk hydrates, which can cause the settling behavior.



(a) (b)

Figure 10. Images of CO_2 hydrate formation during water dropping at 2˚C and 500 psi: (a) water pocket stayed at the bottom of CO_2 liquid at the beginning, (b) CO_2-hydrate crystals at the bottom side of the reactor after 3.45 hours.



(a) (b)

Figure 11. Images of CO_2 hydrate formation during water dropping at 3˚C and 500 psi (near the equilibrium point): (a) a water pocket stayed at the bottom of CO_2 liquid at the beginning, (b) a hydrate-shelled water pocket stayed at the bottom side of the reactor after 12 hours.

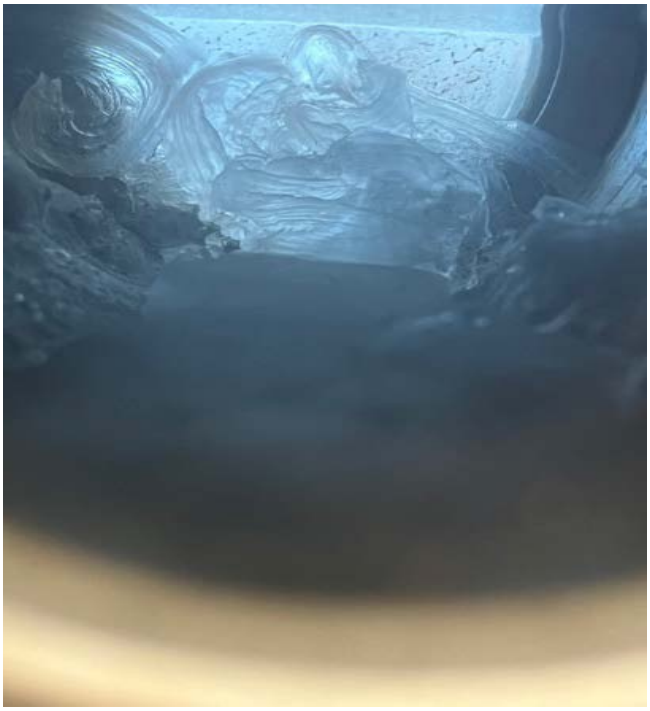


Figure 12. Images of CO_2 hydrate formation from water dropping at 3˚C and 500 psi (near the equilibrium point), CO_2-hydrate crystals on the bottom side of the reactor after 34 hours.

5. Conclusions

Discharging carbon dioxide (CO_2) to seafloors in the form of CO_2-hydrates can potentially store CO_2 in virtually unlimited quantities. Realizing the process requires a thorough understanding of CO_2-hydrate forming behavior in seawater conditions. One of the major concerns is the gravitational stability of the produced bulk CO_2-hydrates. The objective of this study was to seek the optimum procedure for producing bulk CO_2-hydrates that would stay at the bottom of the water due to gravity. This objective was achieved by direct visualization of CO_2-hydrates through the glass windows of a reactor under various pressure and temperature conditions with mixing methods of CO_2-bubbling and water-dropping. The following conclusions are drawn.

1) CO_2 bubbling into the water phase initiates CO_2 hydrates at the surfaces of bubbles, creating bubble shells. The bubble shells reduce further contact between CO_2 and water, hindering the growth of hydrates. The hydrate-shelled CO_2 bubbles move upward due to buoyancy.

2) Reducing pressure causes the collapse of bubble shells. However, the collapsed shells still stay at the interface of water and CO_2, indicating that the collapsed shells are CO_2-rich, *i.e.*, there are still free CO_2 molecules outside of the hydrate structures.

3) Continuous injection of CO_2 to the bottom of the water phase can create hydrate-coated CO_2 channels. The tortuosity of the channels decreases as the CO_2 flow rate increases. Further studies are needed to determine the critical condition

for the change from CO_2 bubbling to CO_2 channeling.

4) Water-dropping into CO_2 liquid initiates CO_2-hydrates at the surfaces of water droplets, creating hydrate-coated water droplets. The hydrate crystals grow outside the hydrate coatings. The bulk hydrates stay at the bottom of the water phase, indicating the gravitational stability of the bulk hydrate.

5) To produce gravitationally stable CO_2-hydrates for deposition on seafloors, CO_2-hydrates should be generated using the process of water spraying into the CO_2 phase, not the process of CO_2-injection into the water phase.

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Author Contributions

Muhammad Towhidul Islam—Experimental investigations. Boyun Guo—Manuscript writing and resources.

Data Availability Statement

The data supporting the findings of this study are included within the article.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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