



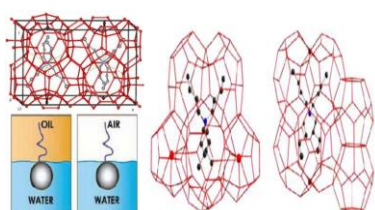
Original Research

Effect of Additives on Hydrate Formation

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GRAPHICAL ABSTRACT



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ABSTRACT

Considering the advantages and disadvantages of hydrate formation, hydrate has always been considered as a problem that should be avoided, and has been considered as an opportunity for revolution in various industries. Solutions have always been proposed to prevent its occurrence and to improve the conditions of its formation. Among the various solutions, the use of additives may be the most appropriate method. In this research study, first, the types of additives and their effect on the formation of hydrates are assessed. Then the results of some recent research in this field are discussed. In general, additives used in the hydrate formation process are divided into two categories: inhibitors and improvers. Injection of chemical inhibitors is a priority over other solutions used to prevent hydrate formation and is widely used in the gas industry. Factors influencing the choice of inhibitors include: cost, degree of poisoning, degree of corrosion, environmental aspects, and bulk density.

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Introduction

Thermodynamic inhibitors affect the thermodynamic equilibrium of the aqueous phase, causing the hydrate equilibrium diagram to shift toward low temperature and high pressure. These substances include alcohols, glycols and salts (they are not used in gas transmission lines due to corrosion by salts). These inhibitors are expensive, destructive to the environment, and highly toxic and volatile.

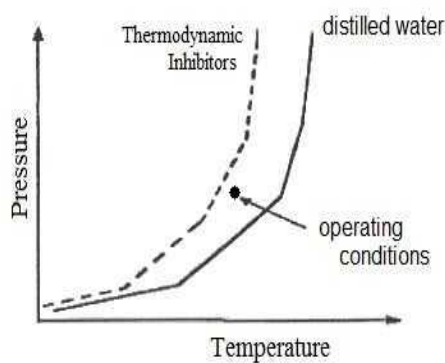


Figure 1. The effect of increasing the thermodynamic inhibitor on the hydrate formation curve.

Synthetic Inhibitors

Kinetic inhibitors delay the growth of crystals and the trapping of hydrocarbons in the ice crystal lattice. The delay time is the time of gas flow in the pipeline up to the moment of hydrate formation in the operating conditions where hydrate formation is possible. The effect of kinetic inhibitors is that they are adsorbed on water molecules and prevent the penetration of gaseous molecules and the formation of their bonds. The most popular synthetic inhibitors are polyvinyl pyrrolidone (PVP), polyvinyl caprolactam (PVCAP),

polymethyl-vinyl lactamide (VIMA), poly acrylpyrrolidine, polymeric compounds of these (VC-7 polymer). Synthetic inhibitors are less expensive to operate than thermodynamic inhibitors, are environmentally friendly and non-toxic.

Takayuki *et al.* [3], Fuzzy equilibrium of clathrite-like hydrate formation in the tetra-n-butyl ammonium bromide system with carbon dioxide and water (in the temperature range of 278.8 to 287.4 with TBAB weight percentage, 0.05 to 0.45 and in pressure of 0.3 MPa, 0.6 MPa and 1 MPa were investigated. The results demonstrated that the equilibrium temperature of clathrite-like hydrates in all three pressures increased with increasing the weight percentage of TBAB for the WTBAB <0.35 and decreased for WTBAB >0.40, and the maximum fuzzy equilibrium temperature was WTBAB = 0.35 and WTBAB = 0.40 was obtained. Lee *et al.* [4] assessed the equilibrium conditions of hydrate formation of a mixture of carbon dioxide and hydrogen gas in the presence of tetrabutyl ammonium bromide and water, and they found that the addition of tetrabutyl ammonium solution with a molar fraction of 0.14% to the gas mixture containing 39% di2.2 carbon monoxide and 60.8% hydrogen causes the pressure of hydrate formation at a temperature of 278.75 K from 11.01 MPa to 3.15 MPa. This indicates a 71.38% reduction in pressure. Also, by increasing the TBAB from the molar fraction of 0.14 to 0.29, the hydrate formation pressure was decreased to 0.85 MPa, indicating a 92.3% decrease in pressure.

They examined the hydrate formation pressure in the molar fraction of 0.21, 0.50, 1, and 2.67 (Figure ??). In addition, at the same concentration of TBAB, the hydrate formation pressure was increased with increasing equilibrium temperature. Lee et al. [4] showed that by increasing the H_2 in the equilibrium pressure mixture, the formation of hydrate enhanced, showing that in a concentration range for TBAB, THF, C_3H_8 and CP additives, tetra-N-butyl ammonium bromide additive the greatest reduction in equilibrium pressure causes the formation of hydrates in the gas mixture.

Chemical enhancers are divided into two groups including, the kinetic enhancers and thermodynamic enhancers.

Kinetic Improvers

Despite the large volume of studies on the benefits of hydrate for use in industry, they have not yet been able to introduce hydrate-based technology that is successful for practical use and in industry.

The biggest problem with this process is the need to find ways to produce hydrate at a high rate. One of the common methods to increase the rate of hydrate formation is to use a stirrer. However, the use of a mixer creates problems that make it difficult to use this method on an industrial scale.

For example, increasing the cost of energy by increasing the thickness of the slurry, filtering and cleaning the reactor, separating the hydrate from the slurry, will increase the capital for the purchase of equipment and also

the cost of repair. Most importantly, in reactors equipped with agitators, system insulation is of great importance.

Therefore, if a method is introduced that mixes the water-gas system at very low agitator speeds or does not require mechanical equipment, there will be a significant reduction in the costs associated with the hydrate formation process. Researchers are using the addition of kinetic enhancers to address these problems. These materials show their improving effect by increasing the solubility of gaseous components in the water phase. Kinetic improvers are divided into two groups: surfactants and hydrotropes.

Surfactants

A group of surface phenomena are created by the properties of soluble molecules called amphiphiles. An amphiphilic molecule is a molecule in which one part tends to dissolve in a particular solvent while another part of it likes another solvent.

These two solvents are immiscible. Therefore, the structure of these molecules is composed of two regions with completely different polarities:

- A group that shows very weak attraction to the solvent (lipophobic group)
- The group that shows a lot of attraction, (Lipophil group)

If one of these solvents is water, then the part of the molecule that likes water is called hydrophilic and the other part that does not like water molecules and likes other

environments such as oily media is called hydrophobic. Amphiphilic molecules are essential in many industrial processes. They are used as detergents, emulsifiers for creams in cosmetics and pharmaceutical industries, etc.

A group of soluble amphiphiles are surfactants. The surfactant is characterized by its tendency to adsorb on the interface.

The surfactant molecule consists of two parts:

- Hydrophilic head (polar or ionic head)
- Hydrophobic tail (non-polar group)

Hydrophilic groups cause the surfactant to dissolve in polar solvents such as water. Hydrophobic groups cause the surfactant to be soluble in non-polar solvents and oils. The shape and size of hydrophilic and hydrophobic groups of surfactants determine many of their characteristics.

Surfactants are classified according to the chemical structure of the head group (hydrophilic) as follows:

- Anionic surfactants: In such materials, the hydrophilic group has a negative charge. Anionic surfactants include alkyl sulfonates ($\text{RNO}_3^- \text{M}^+$) and sodium dodecyl sulfate - (SDS) $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$.
- Cationic surfactants: In such materials, the hydrophilic group has a positive charge. Examples of cationic surfactants include alkyl trimethyl ammonium halides ($\text{RN}^+ (\text{CH}_3)_3 \text{X}^-$) and tetradecyl trimethyl ammonium $\text{CH}_3 (\text{CH}_2)_{13} \text{N}^+ (\text{CH}_3)_3 \text{Br}^-$.

Non-ionic surfactants: In such materials, the hydrophilic group has no electric charge but is

very polar. In fact, the water-soluble component consists of hydroxyl groups with a chain. For example, alkyl poly oxy ethylene ($\text{R} (\text{OCH}_2\text{CH}_2)_n \text{OH}$) fall into this group.

- Two ion surfactants: Materials that contain both positive and negative electrical charges. Long-chain amino acids ($\text{RNa}^+ (\text{CH}_2)_2 \text{CH}_2 \text{CH}_2 \text{SO}_3^-$) fall into this group.

When the concentration of surfactants reaches a certain value, aggregation occurs spontaneously and the molecules of surfactants arrange themselves into regular assemblies known as micelles. Figure 3 shows an illustration of micelle formation.

Depending on the chemical structure and molecules in the system, the micelles can be cationic, anionic, dual ionic or nonionic.

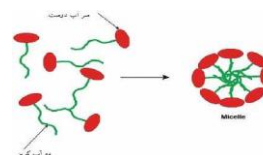


Figure 2. Michel formation

The surfactant must exhibit surface activity, meaning that the surfactant is adsorbed at low concentrations to the joint surface or surface and reduces surface tension. The common surface can be the boundary between liquid and gas phases (air) or the boundary between two immiscible phases (liquid-liquid or liquid-solid).

Surface activity is obtained when the number of carbon atoms in the hydrophobic tail is greater than 8. The appropriate range for the number of carbon atoms in the hydrophobic tail is between 10 and 18. (If this number is

less than 8, the solubility is high; if this number is higher than 18, the solubility is very low). if a surfactant is added to the system, the molecules the surfactant moves towards the joint surface and the hydrophobic tail is placed flat on the joint surface and the hydrophilic head is oriented towards the fuzzy containing the polar material.

The molecules of the surfactant on the surface destroy the gravitational force between the polar and non-polar molecules and replace the polar and non-polar molecules on the surface. The interaction between the hydrophilic head and the polar phase and the hydrophobic tail and the non-polar phase is stronger than the interaction between the polar and non-polar molecules.

Therefore, the interfacial tension decreases. If one of the phases is gas, the term surface tension reduction is used instead of interfacial tension reduction.

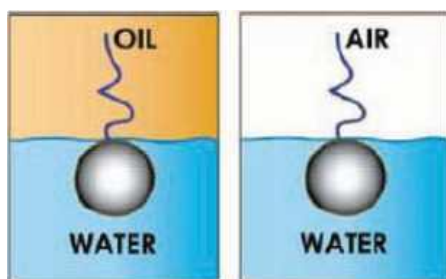


Figure 3. Orientation of hydrophilic head and hydrophobic tail at the common level of different environments

Hydro tropes

Hydro tropes are aromatic compounds found in nature in both plant and animal forms. Hydro tropes consist of a small hydrophobic part and a large hydrophilic part. In fact, hydro

tropes with their short, bulky and compact hydrophobic part are separated from the surface-active substances whose hydrophobic part is longer.

Hydro tropes have the ability to dissolve more non-polar molecules (gases) in water than surfactants. As mentioned earlier, the longer the hydrophobic part, the more surface material the material has, and the shorter the hydrophobic part, the higher the solubility of the gas molecules in the water. In the case of surfactants, the length of this chain is large, so they show surface activity, and in the case of hydro tropes, because the length of this chain is shorter, they have the ability to dissolve more non-polar gas molecules in water. In fact, in surfactants.

As the length of the hydrophobic chain is large, the molecules of this substance due to their greater hydrophobicity and distance from water, tend to be present in the joint, and therefore by attending the joint and according to the mechanism previously explained to them reduces surface tension to a considerable extent compared to hydro tropes. However, the hydrophobic chain length is shorter, preventing the hydro tropes from forming micelles, unlike surfactants (whereas surfactants form spherical or cylindrical micelles. Can create). Therefore, the shape of the micelles is a closed micelle that opens from the outside, which makes non-polar molecules easier to enter the space formed by hydrophobic chains and finally enter through the hydrophilic head.

Melt and dissolve in it and thus can increase the solubility of non-polar molecules such as gaseous hydrocarbon components in water to a significantly greater extent than surfactants. In this way, surfactants increase the rate of hydrate formation by reducing the surface tension or in other words by reducing the mass transfer resistance at the gas-liquid interface, and also by increasing the solubility if the hydrophobic chain length in these materials is small. Gases can also increase the rate of hydrate formation.

Thermodynamic Improvers

One of the costliest steps in hydrate formation is the process of compressing gas to relatively high pressures, as well as the need for low temperatures to form hydrates.

To solve this problem, researchers have done a lot of research on thermodynamic enhancers. Thermodynamic improvers advance the formation of gaseous hydrates to form at low pressures and high temperatures (optimal thermodynamic conditions). Thermodynamic improvers fall into two general categories: water-insoluble compounds and water-soluble compounds. Water-insoluble thermodynamic enhancers, depending on their molecular size, form two types of hydrate structures. Materials such as cyclobutane, cyclopentane, cyclohexane, tetrahydropyrene, and neopentane form structure II.

When gaseous hydrates are formed in the presence of these water-insoluble improvers, due to the large size of these compounds, they

are placed in large cavities and gas molecules occupy small cavities.

In this way, water-insoluble compounds facilitate the thermodynamic conditions of hydrate formation by creating dual hydrates. Due to the insolubility of these compounds in water, the effect of the concentration of these substances on the rate of curing is not significant. Thermodynamics facilitate hydrate formation.

Tetrahydrofuran is one of the most important non-saline thermodynamic enhancers of water-soluble which greatly facilitates the formation of hydrates.

One of the most practical advantages of using this material, which belongs to the family of cyclic ethers, is the storage of hydrogen at low pressures. Hydrogen alone forms hydrate at a temperature of 280 K and a pressure of 3000 bar, while in the presence of THF hydrogen hydrate is formed at a temperature of 279.6 K and a pressure of 50 bar.

Problems such as volatility, toxicity and flammability limit the use of this substance. Salt compounds can significantly facilitate the formation of hydrates and are able to form hydrates in environmental conditions. The nature of the hydrate structure formed in the presence of water-soluble salt compounds differs from the structure of simple gaseous hydrates. In addition to being located inside the cavities, these materials also participate in the structure of hydrate networks. In 1940, the first structures were discovered by Fuller et al. Because tetraalkylammonium salts produce

clathrate-like hydrates, these types of hydrates are also called ionic hydrates.

Clathrate-like hydrates are formed from tetra-n-butyl or tetra-n-amyl salts on quaternary ammonium or phosphonium cations. The anionic part of these compounds forms hydrogen bonds with water molecules and their cationic part occupies the cages.

Tetra-n-butyl-ammonium halide (TBAX), tetra-n-butyl ammonium bromide (TBAB), (TBAC) and tetra-n-butyl ammonium fluoride (TBAF) are some of the salts that make up clathrate-like hydrates.

The most common structures of clathrate-like hydrates for tetra-n-butyl ammonium salts are cubic structure I (CSS-I), quadrilateral structure I (TS-I) and hexagonal structure I (HS-I). Tetra-N-butyl ammonium halide (TBAX) -clathrate hydrates are stable at atmospheric pressure and ambient temperature.

Tetra butyl ammonium bromide (TBAB) is a semi-clathrate hydrate

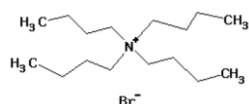


Figure 4. The molecule of tetra n-butyl ammonium bromide with molecular formula $(\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2)_4\text{NBr}$

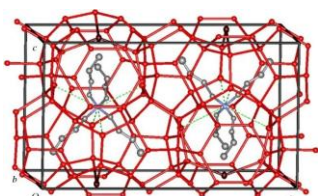


Figure 5. A single cell of tetra butyl ammonium bromide semiclathrate hydrate

Table 1. Geometric Properties of Semi-Clathrate Hydrate

Quaternary-like structure			Hydrate structure
Large cavity	Large cavity	Small hole	The size of the hole
$5^{12}6^3$	$5^{12}6^2$	512	The type of cavity
4	4	6	Number of holes in the structure
52			Number of water molecules in type A.
76			Number of water molecules in type B.

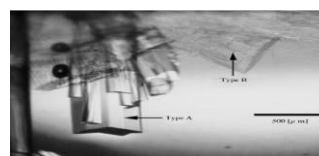


Figure 6. Type A and B hydrates for TBAB without the presence of gaseous components

Three structures have been reported for the tetra-n-butyl ammonium chloride-like hydrates (TBAC): $\text{TBAC} \cdot 32\text{H}_2\text{O}$ and $\text{TBAC} \cdot 30\text{H}_2\text{O}$, $\text{TBAC} \cdot 24\text{H}_2\text{O}$. The stability of each of these structures depends on TBAC concentration, temperature and pressure.

Tetra-N-butyl ammonium fluoride (TBAF) -clathrate hydrate has two known crystalline structures, one of which has a cubic structure (CSS-I) with a hydrate number of 29.7 ($\text{H}_2\text{O}_{29.7}/29$ TBAF).

The crystal unit network of this structure has four fifteen-sided cavities ($5^{12}6^3$), sixteen fourteen-sided cavities ($5^{12}6^2$), ten cavities. It is a small dodecahedron (512) and 164 water

molecule. The stability of the quadrilateral structure is low and this structure usually turns into a cubic structure. Large cavities in these structures are occupied by the tetra-*n*-butyl ammonium cation (TBA⁺) and small cavities are filled or left empty by the guest molecules.

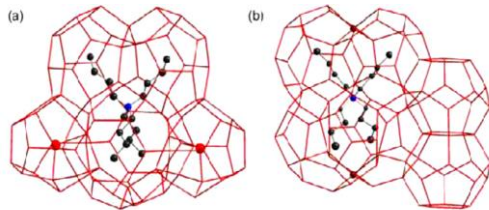


Figure 7. Clathrite-like structures a) H₂O 29.7 TBAF and b) H₂O₃₈ TBAB

Due to the issues raised regarding the gas hydrates, clathrite-like hydrates are more important due to the more suitable thermodynamic conditions of their formation. However, their production process has not been industrialized so far. The main reasons for which are the low storage capacity and slow process.

Conclusion

If only 5% of the hydrated gas tanks can be removed, the amount will reach the predicted amount of gases that can be removed from non-hydrated gas sources. In any case, producing gas from hydrates with conventional technologies is not economical. Hydrate stability zone (HSZ), i.e. the place where gas hydrates are stable, depends on temperature, pressure, ground temperature changes, gas volume, gas percentage composition and thickness (from a few meters

to more than 1000 m). The above factors can also control the depth at which the HSZ is formed. Gaseous hydrates have been identified in the continental shelf sediments around North America, as well as areas with sludge sediments in the North Continents. The US Geological Survey estimates a total of 320,000 trillion cubic feet of gaseous hydrate. In kinetic studies on the formation of gas hydrates, the aim is to investigate parameters such as induction time and storage capacity. In general, kinetic studies in this field with and without improvers are very limited, unlike thermodynamic studies in this field. For this reason, much of the kinetic data for the formation of gaseous hydrates and clathrite-like gaseous hydrates are incomplete. Therefore, the kinetic study of the formation of hydrates, especially pseudo-clathrites, can be the first priority of research in this field.

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