In the early 20th century, the natural gas industry started facing the problem of unexpected pipeline blockage. In 1934, Hammerschmidt showed that blockages appearing to be ice were actually gas hydrates. Clathrate hydrates (or gas hydrates) are nonstoichiometric crystalline water-based solids physically resembling ice, in which small guest molecules, such as methane, ethane, propane, carbon dioxide, and hydrogen sulfide, are trapped inside cavities of hydrogen bonded, frozen water molecules.

Gas hydrates crystallize in three common structures: cubic structures I (sI), cubic structures II (sII), and hexagonal structure H (sH), depending on the nature and the size of the guest molecule. There is a strong relationship between hydrate crystal structure and such properties as phase equilibria and heat of dissociation.

Formation of gas hydrates can lead to serious operational, economic, and safety problems in the petroleum industry due to potential blockage of oil and gas equipment. However, these compounds can have many positive applications, due to their interesting properties such as high storage capacity (STC) and dissociation enthalpy. They can be used for separation, storage and transportation of oil and gas, CO₂ separation and sequestration, concentration of liquid food products, and air conditioning systems.

The presence of semiclathrate hydrate former salts such as tetra n-butylammonium chloride (TBAC) mild the thermodynamic conditions of hydrate formation, considerably. As opposed to the abundant studies undertaken on the thermodynamics of hydrate forming in the presence of these salts, their kinetics needs further investigation. In this research, the kinetics of methane-TBAC semiclathrate hydrate forming has been focused on. The effects of the polysorbate 80, various weight fractions of TBAC, and beginning pressure of the cell on the moles of gas encaged in hydrate cavities and storage capacity of formed hydrates are investigated. The tests were carried out in the isothermal condition of 276.65 K. The results revealed that by raising the weight fraction of TBAC from 5 to 15 wt%, the moles of gas encaged in hydrate cavities and storage capacity of formed hydrate decreases. Utilization of polysorbate with a concentration of 15 ppm along with TBAC, promotes the kinetics of semiclathrate hydrate forming process (HFP) and raises the storage capacity, compared to TBAC aqueous solution. Finally, the influence of the beginning pressure of the cell on the kinetics of TBAC-methane hydrate forming is studied. Results indicate that by raising the beginning pressure of the cell, the moles of gas encaged in hydrate cavities and storage capacity raises.

Keywords
TBAC, Semiclathrate hydrate, Kinetics, Methane, Polysorbate 80

1. Introduction

In the early 20th century, the natural gas industry started facing the problem of unexpected pipeline blockage. In 1934, Hammerschmidt showed that blockages appearing to be ice were actually gas hydrates. Clathrate hydrates (or gas hydrates) are nonstoichiometric crystalline water-based solids physically resembling ice, in which small guest molecules, such as methane, ethane, propane, carbon dioxide, and hydrogen sulfide, are trapped inside cavities of hydrogen bonded, frozen water molecules.

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Formation of gas hydrates can lead to serious operational, economic, and safety problems in the petroleum industry due to potential blockage of oil and gas equipment. However, these compounds can have many positive applications, due to their interesting properties such as high storage capacity (STC) and dissociation enthalpy. They can be used for separation, storage and transportation of oil and gas, CO₂ separation and sequestration, concentration of liquid food products, and air conditioning systems.

Gas hydrates are formed when gas molecules with appropriate size and water molecules come into contact under favorable thermodynamically conditions, that is high pressure and relatively low temperature. Therefore, moderating the hydrate formation conditions is essential to benefit their positive applications. Promoting of kinetics and moderating of thermodynamics of gas hydrate formation have been focused on by many researchers in recent years. In 1940, Fowler et al. discovered a new structure of the hydrates, called semiclathrate hydrates, which were formed by tetra n-butylammonium, tetra n-butyl phosphonium, and tri n-butyl sulphonium salts. Semiclathrate hydrate formers such as tetra n-butylammonium chloride (TBAC) mild the thermodynamic conditions of hydrate formation, considerably.
nium chloride (TBAC), tetra n-butylammonium bromide (TBAB), and tetra n-butylammonium fluoride (TBAF), moderate the thermodynamic conditions of gas hydrate formation, considerably\(^\text{40,41,43}\). Mohammadi and coworkers investigated the influence of various weight fractions TBAC and TBAF on methane, carbon dioxide, and nitrogen phase diagram. They showed that the addition of these thermodynamic promoters by shifting the p-T curves of the double gas + TBAC/TBAF semiclathrate systems to the low pressure and high temperature regions, considerably, moderate the thermodynamic conditions of gas hydrate formation\(^\text{40,41}\). In another research, Kamran-Pirzaman and coworkers measured the dissociation data of methane/carbon dioxide hydrate formation in the presence of TBAC and TBAF. They concluded that both of these additives, noticeably moderate the thermodynamics of methane/carbon dioxide hydrate formation\(^\text{46}\). Thermodynamics of hydrate formation in the systems containing tetra n-butylammonium salts is widely studied in recent years but the kinetics of these systems need further investigation. In 2016, Mech and coworkers studied the kinetics of methane hydrate formation in the presence of tetrahydrofuran (THF), TBAB, and sodium dodecyl sulfate (SDS). Their results showed that simultaneous utilization of THF and TBAB at low pressures and low weight fractions of TBAB promotes the kinetics of gas hydrate formation\(^\text{47}\).

In this research, the influence of an environmentally friendly thermodynamic additive, TBAC with concentration of 5 wt% and 15 wt%, and a nonionic surfactant, polysorbate 80 with concentration of 15 ppm is investigated on the moles of gas encaged in hydrate cavities and STC of methane hydrate. We, also, studied the influence of beginning pressure on the kinetics of methane hydrate formation in the presence and absence of TBAC and polysorbate 80.

2. Experimental

2.1. Materials and Setup

Table 1 shows the used materials and their purities and suppliers. The schematic image of the setup used in this work is shown in Fig. 1\(^\text{48,49}\). The reactor is made of stainless steel with an effective volume of 169 cm\(^3\). It has two needle valves withstands up to 20 MPa for injecting and discharging the gas, and two ball valves for loading and depletion of the solution. A circulator containing water and ethylene glycol with a volume ratio of 1/3 was employed to keep the reactor temperature at a desired value during the tests. A rocking-cell system was employed to mix the solution inside the reactor. The temperature and pressure of the reactor were monitored continuously along the time during the experiments by means of a thermocouple with an accuracy of 0.1 K and a pressure transducer with an uncertainty of 0.01 MPa. A control panel acquires the temperature and pressure of the reactor and then feeds to the computer. The panel records the obtained data using an in-house software at every 10 sec.

2.2. Procedure

We reported the procedure in our previous works\(^\text{48,49}\). At the first step of each experimental run, the reactor was washed with water for 15 min and then rinsed and eluted properly with distilled water. To evacuate inside the reactor, a JB Industries’ PLATINUM series vacuum pump was employed. Thereafter, 50 cm\(^3\) aqueous phase sample was prepared and filled into the reactor.

By adjusting the circulator temperature to 276.65 K, the methane gas was injected into the reactor to the considered pressure (7 MPa or 9 MPa), and the electromotor was also turned on with speed of 10 rpm. By
consumption of methane due to the forming of gas hydrate, the pressure of the reactor was reduced. The temperature and pressure data were recorded by a computer in desired time interval.

3. Results and Discussion

In this research we have studied the effect of TBAC and polysorbate 80 on the kinetics of methane hydrate formation. Aqueous solutions of (a) TBAC with weight fractions of 5 wt% and 15 wt%, (b) polysorbate with a concentration of 15 ppm, and (c) TBAC (15 wt%) + polysorbate (15 ppm) were prepared, in order to study the kinetics of TBAC-methane hydrate forming. All experiments were carried out at a temperature of 276.65 K in a stirred batch reactor. Since the formation of methane hydrate in the absence of TBAC is occurred in the relatively high pressures and low temperatures, we have chosen these conditions to compare the formation of methane hydrate in the presence and absence of used additives.

To calculate the moles of methane encaged in hydrate cavities during hydrate forming process (HFP), Peng-Robinson equation of state50) was used. The following equation is employed to evaluate the moles of gas encaged in hydrate cavities during HFP51).

\[ \Delta n_{CH_4} = \frac{P_0 V_0}{Z_0 R T_0} - \frac{P V_t}{Z_t R T_t} \]  

(1)

where \( P \) and \( T \) respectively, are the pressure and temperature of the reactor. \( V \) is the volume of the gas inside the reactor; \( Z \) is the compressibility factor calculated by the Peng-Robinson equation of state50); \( R \) is the universal gas constant; and subscripts 0 and \( t \) stand for conditions of the reactor at time \( t = 0 \) and time \( t \), respectively.

Gas uptake curves of methane-TBAC HFP in the presence of various weight fractions of TBAC are illustrated in Figs. 2 and 3. As can be seen in these figures, using TBAC with a weight fraction of 5 wt%, raises the moles of gas encaged in hydrate cavities at both beginning pressures of 7 MPa and 9 MPa. While utilization of 15 wt% TBAC decreases the moles of gas encaged in hydrate cavities per mole of water during hydrate forming, compared to distilled water. The moles of methane encaged in hydrate cavities per mole of water in aqueous solution of TBAC with weight fractions of 0, 5, and 15 wt%, respectively, are 49.85, 58.72, and 42.04 mmol/mol, at beginning pressure of 7 MPa. That means the moles of methane encaged in hydrate cavities per mole of water in the presence of 5 wt% TBAC is 17.8 % more than that of distilled water; and the moles of methane encaged in hydrate cavities per mole of water in the presence of 15 wt% TBAC is 15.7 % less than that of distilled water.

The structure of hydrates are formed in the presence of TBAC are different from the common hydrate structures (sI, sII, and sH)52),53). The Cl– ions of TBAC participate in the lattice structure and TBA+ ions occupy the large cages of formed hydrates. Therefore, these new structures are called semicloathrates. The presence of TBAC plays a dual role in HFP. This compound by shifting the phase diagram of methane hydrate forming to the stabilized regions41),43),54), and therefore, raising the driving force of gas hydrate forming, promotes the kinetics of methane hydrate forming and by changing the structure of gas hydrates to semicloathrates and occupying the large cages by TBA+ ions, inhibits the kinetics of gas hydrate forming.

As illustrated in Figs. 2 and 3, at low weight fractions of TBAC (5 wt%), the positive influence of this compound dominates on its negative effect. By raising the weight fraction of TBAC from 5 to 15 wt%, the number of cavities occupied by TBA+ ions raise, and therefore, the number of left cages occupied by gas molecules decrease that causes the decrease of the moles of gas encaged in hydrate cavities during HFP.

Meanwhile, the electrostatic forces between the TBAC ions and the ions of water molecules (H+ and OH–) affect the interactions of water and gas molecules (van der Waals forces), resulting in the decrease of gas consumption. The lower activity of aqueous solution containing TBAC in contrast to pure water is another reason for the inhibition effect of TBAC aqueous solu-
Similar behavior was observed for the solutions containing polysorbate. The influence of TBAC on the moles of gas encaged in hydrate cavities in the presence of polysorbate with a concentration of 15 ppm is shown in Figs. 4 and 5. Utilization of TBAC with a weight fraction of 15 wt% along with polysorbate with a concentration of 15 ppm, by occupying of large cages, decreases the quantity of gas uptake, compared to polysorbate aqueous solution.

The influence of polysorbate on the moles of gas encaged in hydrate cavities per mole of feed water during HFP in the presence and absence of TBAC is depicted in Figs. 6-9. As shown in Fig. 6, at beginning pressure of 7 MPa, the moles of gas encaged in hydrate cavities for distilled water and solution of polysorbate with a concentration of 15 ppm are 49.85 mmol/mol and 60.95 mmol/mol, respectively. At this beginning pressure, the quantity of gas uptake for the systems of TBAC (15 wt%) and TBAC (15 wt%) + polysorbate (15 ppm), respectively, are 42.03 mmol/mol and 44.75 mmol/mol. The results indicate that, the experiments carried out in the presence of 15 ppm polysorbate have higher moles of methane uptake per mole of water as compared with that samples in the absence of polysorbate. Polysorbate is a non-anionic surfactant that
when dissolved in water, by decreasing the surface tension of the solution, decreases the resistance of water molecules at the surface, and therefore raises the moles of gas encaged in hydrate cavities.

The influence of beginning pressure of the cell on the moles of gas encaged in hydrate cavities during the process of hydrate forming is investigated in this section. The gas uptake profiles for the systems of water + methane, water + methane + TBAC, water + methane + polysorbate, and water + methane + TBAC + polysorbate is plotted in Figs. 10-14. The moles of gas encaged in hydrate cavities noticeably raises with a raise in the beginning pressure of the cell. The moles of gas encaged in hydrate cavities in distilled water at beginning pressures of 7 MPa and 9 MPa are 49.85 mmol/mol and 87.21 mmol/mol of water. This means that by raising the beginning pressure of the cell from 7 to 9 MPa, the moles of gas encaged in hydrate cavities raises about 74.9 %. As observed in Figs. 10-14, raising the beginning pressure of the cell could effectively promotes the kinetics of methane-TBAC HFP. By raising the beginning pressure of the cell, the driving force of HFP raises that causes the raise of the moles of gas encaged in hydrate cavities.

STC is a main kinetic parameters of hydrate forming that is calculated based on the theoretical calculation presented in our previous publication51).

$$\text{STC} = \frac{V_{\text{STP}}}{V_{\text{H}}} = \frac{\Delta n_{\text{CH}_4} R_{\text{STP}}}{P_{\text{STP}}} / \frac{V_{\text{H}}}{V_{\text{H}}}$$  \hspace{1cm} (2)

In Figs. 10-14, we have compared the kinetics of gas uptake in the presence and absence of TBAC and polysorbate 80. In these figures we tried to show the effect of used additives on the kinetics of gas uptake. The values of gas uptake used in Eq. (2) were determined at $t = 1400$ min.

Figure 15 presents the comparison of the amount of STC of hydrates for different studied samples. These data are listed in Table 2.

As can be found from Fig. 15 and Table 2, using
low weight fraction of TBAC (5 wt%) raises the STC of formed hydrate at both beginning pressures of 7 MPa and 9 MPa. The amount of STC at beginning pressure of 9 MPa for the systems of water + methane and water + methane + TBAC (5 wt%) are 106.09 v/v and 122.70 v/v, respectively. That means the amount of STC in the presence of 5 wt% TBAC is about 15.7 % higher than that of distilled water. Raising the weight fraction of TBAC to 15 wt% causes a decrease in the amount of STC for both beginning pressures of 7 MPa and 9 MPa. At beginning pressure of 9 MPa, the amount of STC for aqueous solution of TBAC with a concentration of 15 wt% is 97.04 v/v. This means that the amount of STC of this solution is about 8.5 % less than that of distilled water.

As previously stated, the presence of TBAC plays a dual role in HFP. Low weight fractions of this compound (5 wt%) by shifting the phase diagram of methane hydrate forming to the stabilized regions, and therefore by raising the driving force of hydrate forming, raise the STC of hydrate. At high weight fractions of TBAC (15 wt%), number of large cages occupied by TBA⁺ ions raise that causes a decrease in the amount of STC.

Utilization of polysorbate with a concentration of 15 ppm presented a positive effect on the STC of formed hydrate at both beginning pressures of 7 MPa and 9 MPa. At beginning pressure of 9 MPa, the amount of STC for the system of methane + water + polysorbate (15 ppm) is 112.56 v/v. Comparing this amount with that of distilled water shows that utilization of 15 ppm polysorbate raises the STC of hydrate forming up to 6 %. Similar behavior is observed for the solutions containing TBAC with a weight fraction of 15 wt%. In fact, the presence of polysorbate by decreasing the surface tension of the solution, enhances the amount of STC of formed hydrate.

Investigating the influence of the beginning pressure of the cell on the STC of formed hydrate shows that, raising the beginning pressure of the cell from 7 to 9 MPa, enhances the amount of gas storage for all systems. For the system of water + methane + TBAC (15 wt%) + polysorbate (15 ppm) at beginning pressures of 7 MPa and 9 MPa, the amounts of STC are 54.44 v/v and 104.92 v/v, respectively. This means that raising the beginning pressure of the cell from 7 to 9 MPa, raises the amount of STC up to 93 %.

Raising the beginning pressure of the cell raises the driving force of hydrate forming and therefore, raises the amount of STC of hydrate forming.

4. Conclusions

Gas hydrate forming in the presence of TBAC was investigated in this research. The influence of the presence of polysorbate, various weight fractions of TBAC, and beginning pressure of the cell on the kinetics of hydrate forming was studied.

Utilization of low weight fractions of TBAC (5 wt%) by shifting the phase diagram of hydrate forming to the

| Additive                  | P [MPa] | Storage capacity [v/v] |
|--------------------------|---------|------------------------|
| Distilled water          | 7       | 60.6                   |
| TBAC (5 wt%)             | 7       | 71.4                   |
| TBAC (15 wt%)            | 7       | 51.1                   |
| Polysorbate (15 ppm)     | 7       | 74.2                   |
| Polysorbate (15 ppm) + TBAC (15 wt%) | 7 | 54.4 |
| Distilled water          | 9       | 106                    |
| TBAC (5 wt%)             | 9       | 123                    |
| TBAC (15 wt%)            | 9       | 97.0                   |
| Polysorbate (15 ppm)     | 9       | 113                    |
| Polysorbate (15 ppm) + TBAC (15 wt%) | 9 | 105 |

a) The maximum uncertainty in the measured storage capacity is expected to be 4 v/v.
stabilized regions, promotes the kinetics of hydrate forming and raises the moles of gas encaged in hydrate cavities and STC. While, at high weight fractions of TBAC (15 wt%), a lot of large cages are occupied by TBA+ ions and therefore the moles of gas encaged in hydrate cavities and STC decreases, compared to distilled water.

The influence of polysorbate on the kinetics of hydrate forming was investigated. The results showed that using this compound with a concentration of 15 ppm, by decreasing the surface tension of the solution, enhances the kinetics of HFP for all samples.

Investigation of the influence of beginning pressure of the cell on the kinetics of HFP showed that raising the beginning pressure of the cell from 7 to 9 MPa, by raising the driving force of hydrate forming, enhances the moles of gas encaged in hydrate cavities and STC.

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