

## Investigation of the kinetics of formation of Clatrit-like dual hydrates TBAC in the presence of CTAB

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### Abstract

As it is always necessary to design a pipeline at high pressure (high density), pipelines are also exposed to ambient temperatures and are usually exposed to low temperatures. On the other hand, in the presence of water vapor (almost all natural gases have some water vapor), and more importantly, the presence of hydrocarbons causes hydrate crystals to form. In this work, the capacity of carbon dioxide hydrate storage in water in the presence of surfactants at different temperatures, pressures and concentrations of TBAC and CTAB additives was calculated and measured using induction time measurement. The results of experiments show that with increasing pressure and decreasing temperature the storage capacity of CO<sub>2</sub> in hydrate increases. Addition of CTAB also dramatically increases the storage capacity, while increasing pressure has a greater impact on the storage capacity of carbon dioxide in the hydrate. The effect of TBAC and CTAB surfactant on the induction of hydrate formation and carbon dioxide storage capacity was investigated. Design Expert software was used to design the experiment. Finally, statistical analysis of the effective parameters on the time of induction of hydrate formation showed that TBAC can decrease the time of induction of hydrate formation compared to other additives. In investigation of the effect of variables on the storage capacity of carbon dioxide gas, it can be concluded that increasing the amount of CTAB surfactant and pressure has the most impact on the increase of carbon dioxide storage capacity compared.

**Keywords:** TBAC; gas hydrate; surfactant; induction time.

### Introduction

On gas transmission lines, there are usually four major factors required for hydration generation. Hydrate generation in addition to partial or complete blockage of gas transmission lines causes the deposition and clogging of distillation trays, nozzles, valves and heat exchangers, abrasion of turbochargers and more. After the discovery of gas hydrates as a cause of obstruction of the gas transmission

pipelines by Hummer Schmidt in 1934, studies in the field of the thermodynamic data collection of hydrates were further developed.

1) Carbon dioxide is mainly produced by combustion of fossil fuels in homes and cars and burning off gases in the burners of refineries and petrochemical industries as well as in thermal power plants and enters the atmosphere and it is one of the effective pollutants in the

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greenhouse effect that causes global and gradual warming. Separation and storage of greenhouse gas is one of the most fundamental environmental issues today [1-3]. One of the methods of carbon dioxide separation and storage is the use of the phenomenon of hydration, which has attracted the attention of many researchers. Carbon dioxide depletion in Deep-sea is one of the solutions that scientists have found to reduce carbon dioxide emissions, instead of releasing carbon dioxide into the air as deep-water hydrates. In this method, carbon dioxide gas is injected at a depth below 400 m and it is trapped by solubilization in water and, due to low temperature and high pressure, deep water carbon dioxide is converted into hydrate at 500 to 900 m above sea level. In this study, the effect of additives (nanoparticles and surfactants) on thermodynamics and kinetics of carbon dioxide hydrate formation is studied and investigated [4-7].

Mr Varaminian and his colleagues investigated the effects of two anionic surfactants (SDS) and (SDBS) and two non-ionic surfactants Triton and Tween 40 on the rate of propane hydrate formation and the results show that SDS at the tested concentrations did not affect the rate of hydrate formation but even at higher concentrations reduced the hydrate formation. SDBS has a significant effect on reducing propane hydrate formation time. At 100 ppm, concentration and stirring speed of 500 rpm reduced propane hydrate formation time to 74%. Triton, like SDS, did not affect the rate of hydrate formation at the tested concentrations. Tween 40 also has a large effect on reducing the propane hydrate formation time, given that it initially reduces the initial rate of propane hydrate formation, but ultimately reduces the propane hydrate formation time.

Moradi and colleagues investigated the effect of two ionic liquids 1-Butyl-3-methyl [BF<sub>4</sub>] and 1-Butyl-3-methyl imidazolium methyl sulfate [Bmim] [MS] on the kinetics of carbon dehydrate formation. The results of their studies showed that increasing these two ionic liquids at a mass concentration of 0.6% at initial pressure 35 bar, by reducing the nuclearisation and induction time, accelerated the process of carbon dioxide formation and acted as a kinetic enhancer. As a result, the coefficients of improvement were 68% and 52%, respectively [8].

Lee and Zhang investigated the kinetics of carbon dioxide hydrate formation in a discontinuous batch reactor in the presence of silicon pentane (CP), as a thermodynamic enhancer, at 274 K and a pressure of 1.9-3.4 KPa. Their experiments were performed in two volumes of 100 and 200 cm<sup>3</sup> of water. The results indicated that the rate of hydrate growth also depended on the pressure in addition to severe dependence on the volume of water. In this study, at the hydrate formation process and the molar ratio of CO<sub>2</sub> trapped to CO<sub>2</sub> in dual hydrates is 100 cm<sup>3</sup> greater than 200 cm<sup>3</sup> water. Increasing pressure to 3.0 MPa also increases the rate of hydrate growth, but with increasing pressure from this value the growth rate decreases [9-12].

Due to the lack of information on the kinetic parameters of quaternary hydrate formation in this study, the effect of addition of Tween and TBAB on the kinetic parameters of carbon dioxide hydrate formation has been investigated. 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 enhancers are very limited, unlike thermodynamic studies. For this reason,

many kinetic data are incomplete for the formation of gas hydrates and quaternary-like hydrates. Therefore, kinetic study of hydrates formation, especially quaternary, can be a top research priority.

### The experiment

Materials used in the experiments include:

1. Carbon dioxide gas
2. Tetra N-butyl ammonium chloride
3. Deionized water

Carbon dioxide gas: The carbon dioxide gas used has a purity of 99.99%.

Tetra-N-Butyl-Ammonium Chloride: Sigma-Aldrich Company's tetra-n-butyl-ammonium chloride was added to the system as a solvent and hydrate formation analyzer with defined concentrations.

Cetyl try methyl ammonium bromide: Cetyl try methyl ammonium bromide  $C_{19}H_{42}BrN$  A model of a cationic surfactant in aqueous solutions is added as another additive for the analysis of hydrate formation considered Soluble.

DM Water: Demineralized water was used to prepare the soluble.

Apparatus: In this study, a device for the formation of hydrates was used. The operating conditions of the device are high pressure and low temperature and all the parts are made of carbon steel that are processed and recorded by connecting temperature and pressure sensors to the computer at specified times. In order to reduce the energy dissipation of the reactor, hydrate formation and all refrigerant fluid transfer fittings and pipes are well insulated. To measure the temperature inside the reactor, a Pt-100 platinum temperature sensor with accuracy of  $\pm 0.1$  K was used. The tank pressure was measured with a BD sensor with an accuracy of about 0.01 MPa. A stir mixer was used to create a proper mixing in the main hydrate reservoir and a pump was used to create a vacuum inside the reactor.

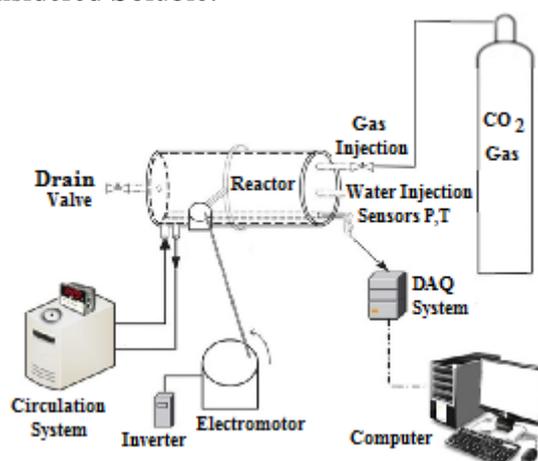


Figure 1. Schematic of the hydrate system

Data acquisition system, transfer and record information: The generated electrical signal by the pressure and temperature sensors is converted to a computer-readable binary number by the DAQ system. The DAQ system, which is more accurate than the sensors,

collects data from the sensors and makes information available for recording, monitoring and controlling the system in an appropriate (digital) form. The DAQ system used in this machine is made by Vista Company. After the data is converted to digital information by the

DAQ, and addressed, the information is transferred to the computer *via* an RS-232 port (serial port). The hardware connection of the DAQ card requires software to deliver and store the necessary information to the user. In this machine, the input data to the computer is stored by the third version of Figure View software. In this software, in addition to displaying data at any time and also displaying data in Figures (temperature-time and pressure-time) for both the absorption tank and the loading tank, data can be registered at intervals (From a second to a few minutes). This software also has the capability to prepare data for analysis in Excel or other statistical software [13-17].

#### **The method of conducting experiment**

The most important goals in kinetic experiments are to obtain parameters such as induction time, storage capacity, water to hydrate conversion, hydration number and hydrate growth rate constant. Generally, the kinetic experiments of hydrate crystal production are performed at constant temperature against thermodynamic experiments. In this study, after adjusting the initial conditions and keeping the temperature constant, the CO<sub>2</sub> gas is injected at a specified average pressure, the gas absorbed into the water and consumed to produce hydrate crystals is not compensated, and thus the system pressure is constantly lowered. And due to the three-phase equilibrium curve of the hydrate, with decreasing pressure, the amount of driving force is also reduced. In all experiments, the system temperature and pressure are recorded at 20-second intervals. In each experiment, after the pressure changes within the reactor are negligible over time (0.05 bar/hr), it is assumed that hydrate formation is completed. In all experiments, the

hydrate production reactor is first washed with distilled water and then discharged into the air using a vacuum pump. The reactor was also washed at the end of the experiment to prevent memory effects in the experiments.

#### **Theory and computation**

The calculation of mole carbon dioxide gas was made using Peng–Robinson equation of state to calculate the number of moles consumed:

$$P = \frac{RT}{v-b} - \frac{a}{v(v+b)+b(v-b)} \quad (1)$$

To do the calculations, we have to rewrite the equation of state in terms of the Z-compressibility coefficient, which is obtained using the Peng–Robinson equation of state (Z-2) [18].

$$Z^3 - (1-B)Z^2 + (A-2B-3B)Z - (AB-B^2-B^3) = 0 \quad (2)$$

$$A = \frac{a\alpha P}{R^2 T^2} \quad (3)$$

$$B = \frac{BP}{RT} \quad (4)$$

$$a = 0.457235 \frac{(RT_c)^2}{P_c} \quad (5)$$

$$b = 0.077796 \frac{RT_c}{P_c} \quad (6)$$

$$\alpha = \frac{[1 + (0.37464 + 1.54226\omega - 0.26992\omega^2)(1 + T_r^{0.5})]^2}{(1 + T_r^{0.5})^2} \quad (7)$$

$$T_r = \frac{T}{T_c} \quad (8)$$

In the above equations, *v* is the molar volume, *T<sub>c</sub>* and *P<sub>c</sub>* are the temperature and pressure at the critical point, respectively, *R* is the global constant of the gases, and *ω* is the decentralization coefficient. By solving the equation in the vapor-liquid calculations, the largest value *Z* is obtained for the vapor phase and the lowest value for the liquid phase is used. Using the *Z* obtained at the initial and equilibrium temperature and pressure, the number of primary and end moles (equilibrium) is obtained [19]:

$$n_0 = \frac{P_0 V_0}{Z_0 R T_0} \quad (9)$$

$$n_e = \frac{P_e V_e}{Z_e R T_e} \quad (10)$$

$$\Delta n = n_0 - n_e \quad (11)$$

$x =$  (Number of moles absorbed)/(Kg of solvent) (12)

$x =$  (Standard volume)/ (Solvent volume /cm<sup>3</sup>) (13)

The relation (12) defines solubility in terms of mole of carbon dioxide to kg of solvent ratio. To calculate the amount of dissolved carbon dioxide in the solvent using the volumetric method, the equation (13) is used, in which the standard volume is calculated by the following equation:

$$V_{STP} = \frac{nRT}{P} \quad (14)$$

In equation (14), the volume of carbon dioxide gas is calculated standard conditions (*i.e.* atmospheric pressure and 25 ° C).

$P_0$ : Gas initial pressure, in terms of (bar)

$P_e$ : Gas equilibrium (final) pressure, in terms of (bar)

$R =$  Global constant of gases (83.14 bar.cm<sup>3</sup>/mol.k)

$T$ : Temperature, in terms of (K)

$Z_0$ : Gas compressibility coefficient at initial pressure

$Z_e =$  Gas compressibility coefficient at final pressure

$V_0$ : Initial volume of gas in cm<sup>3</sup>

$V_e$ : Final volume of gas in cm<sup>3</sup>

Calculate storage capacity

$$SC = \frac{V_{STP}}{V_H} = \frac{\Delta n CO_2 R T_{STP} / P_{STP}}{V_H} \quad (15)$$

In the above equation, the storage capacity is calculated in terms of the standard volume of carbon dioxide gas

to the volume of hydrate gas. In equation (6), pressure, temperature and global constant of gases are considered in standard conditions. In this equation,  $V_H$  is the volume of hydrate obtained. (Here considered 100 cm<sup>3</sup>).

### Statistical analysis of results obtained from experimental tests

In this project, Design Expert software version 11 is used to design the experiment. Response surface experiment design method and Central Composite method are used for statistical analysis. In this case, four parameters affecting the process of carbon dioxide hydration in water are as follows: Pressure ranges from 40bar to 50bar, temperatures from 275K to 279K and additives ranging from 0 to 3wt%. The software has randomly designed 21 experiments, which will be discussed later. As mentioned, the four parameters are the A temperature of the reactor temperature. Parameter B is reactor pressure, parameter C is TBAC concentration and parameter D is CTAB concentration. The result of changes in the above parameters on the R1 factor induction time of hydrate formation in the presence of nanoparticles and surfactants were studied and recorded lab-form. In this paper, the statistical analysis of the effect of regulatory parameters on induction time of hydrate formation is investigated. Values were determined in vitro. These values are given in Table (1).

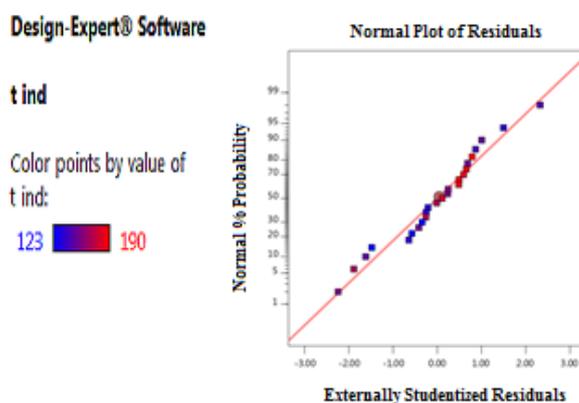
In Table (1), as can be seen, the software has designed randomly 21 experiments. The amount of induction time of hydrate formation must be obtained for each and with the designed conditions and the table has to be fulfilled [20-22].

**Table 1.** Results of experimental experiments to determine the time of induction of hydrate formation

Run	Build Type	Factor 1	Factor 2	Factor 3	Factor 4	Response 1
		A:Temperature	B:Pressure	C:TBAC	D:CTAB	t <sub>ind</sub>
		k	bar	Wt%	Wt%	Sec
1	NA	277	45	1.5	0	170
2	NA	279	40	0	3	173
3	NA	277	40	1.5	1.5	170
4	NA	277	45	1.5	1.5	168
5	NA	279	50	3	0	163
6	NA	277	50	1.5	1.5	165
7	NA	277	45	1.5	1.5	168
8	NA	277	45	1.5	3	165
9	NA	275	50	0	3	168
10	NA	275	40	0	0	177
11	NA	277	45	1.5	1.5	168
12	NA	279	45	1.5	1.5	168
13	NA	277	45	3	1.5	163
14	NA	277	45	1.5	1.5	168
15	NA	275	45	1.5	1.5	167
16	NA	279	40	3	3	163
17	NA	277	45	0	1.5	173
18	NA	275	40	3	0	167
19	NA	279	50	0	0	173
20	NA	277	45	1.5	1.5	168
21	NA	275	50	3	3	158

**Table 2.** Response statistics table

Std. Dev.	5.19	R <sup>2</sup>	0.9863
Mean	154.72	Adjusted R <sup>2</sup>	0.9861
C.V. %	3.36	Predicted R <sup>2</sup>	0.9856
		Adeq Precision	32.7659



**Figure 1.** Data normal dispersion figure

In this section, the effect of each of the defined parameters on the induction time of hydrate formation is discussed.

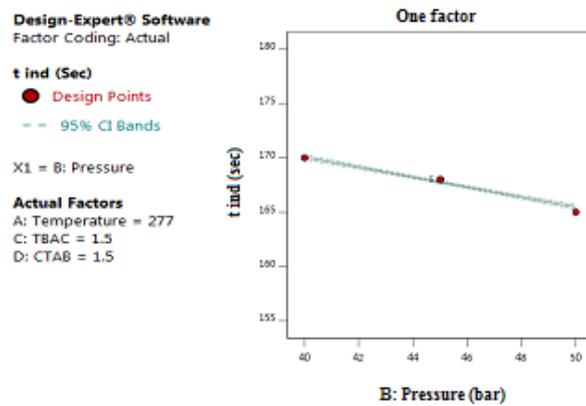


Figure 2. Effect of pressure on time induction of hydrate formation

In this system, according to Figure (2), the increase in pressure has a decreasing effect on the amount of time

induction, which is a positive effect on the formation of hydrates.

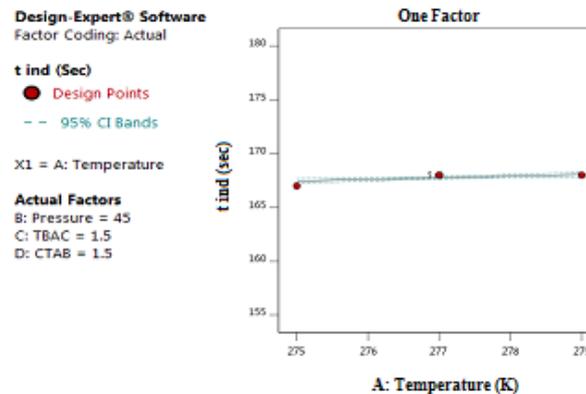


Figure 3. Effect of temperature on induction time of hydrate formation

According to Figure (3), temperature changes have an additive effect on that temperature range. This effect is a negative effect on hydrate formation.

Adding TBAC to the system reduces the induction time of hydrate formation (Figure 4) [24].

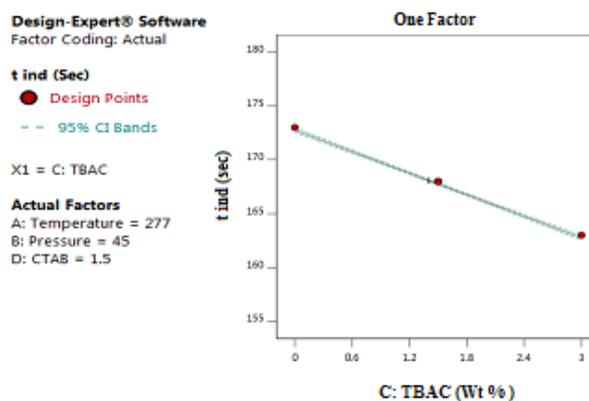


Figure 4. Effect of TBAC on induction time of hydrate formation

Adding CTAB surfactant to the system results in reducing induction time. Comparing the two Figures (4 and

5) shows that the surfactant's effect on reducing induction time is less than that of TBAC [25].

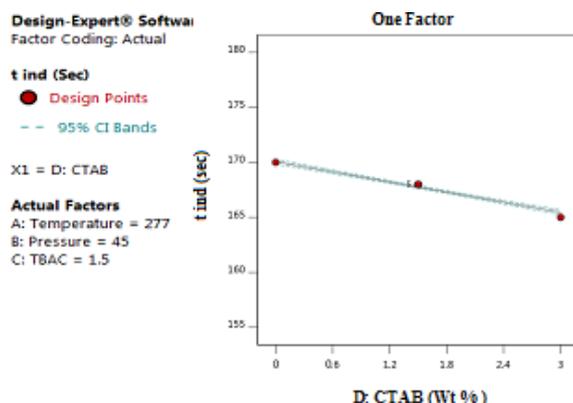


Figure 5. Effect of CTAB on induction Time of Hydrate Formation

In comparison of these two Figures, the effect of TBAC surfactant relative to CTAB on reducing induction time can be observed. What can be investigated is that at a constant temperature and pressure with each of the additives mentioned above, the induction time decreases. As previously mentioned, Design Expert software version 11 is used to design the experiment. Response

surface experiment design method and Central Composite method are used for statistical analysis. For each experiment performed at different temperatures and pressures and different surfactant concentrations, the values were determined in laboratory. These values are given in Table 3 [26].

Table 3. Results of experiments to determine storage capacity

Run	Build Type	Factor 1	Factor 2	Factor 3	Factor 4	Response 1
		A:Pressure	B:Temperature	C:TBAC	D:CTAB	SC
		bar	k	Wt%	Wt%	
1	NA	50	279	3	3	199
2	NA	50	279	0	3	195
3	NA	50	275	0	3	188
4	NA	50	275	0	0	160
5	NA	45	279	1.5	1.5	149
6	NA	40	275	0	0	90
7	NA	40	279	3	0	103
8	NA	45	277	1.5	0	131
9	NA	40	275	3	3	128
10	NA	40	275	0	3	120
11	NA	50	275	3	0	166
12	NA	45	277	1.5	3	162
13	NA	45	277	0	1.5	143
14	NA	40	279	0	0	94

In Table 4, the detection coefficient ( $R^2$ ) shows the concordance of the experimental data with the predicted data indicating that the closer this

number is to one, the better the matching. According to the numbers shown, the experimental data are in excellent agreement.

**Table 4.** Response statistical information table

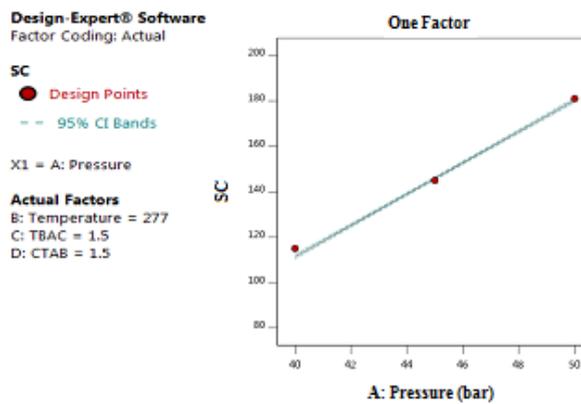
Std. Dev.	2.83	R <sup>2</sup>	0.9856
Mean	77.22	Adjusted R <sup>2</sup>	0.9823
C.V. %	3.66	Predicted R <sup>2</sup>	0.9789
		Adeq Precision	78.4670

**Investigating the effect of variables on storage capacity**

In this section, we examine the effect of each of the defined parameters on the carbon dioxide storage capacity.

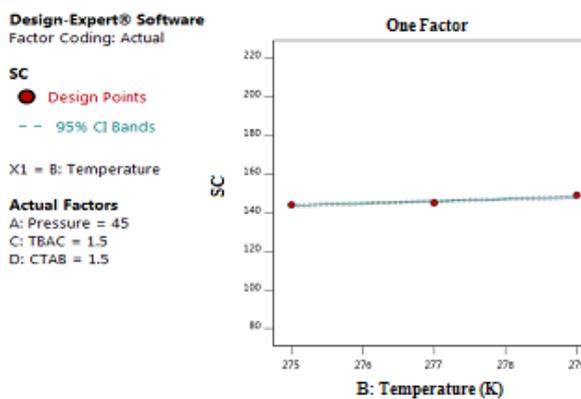
**Investigating the effect of variables on storage capacity**

In this section, we examine the effect of each of the parameters defined on the carbon dioxide storage capacity.



**Figure 6.** Effect of pressure on storage capacity

In this system, according to Figure 6, the amount of storage capacity increases dramatically with increasing pressure.



**Figure 7.** Effect of temperature on storage capacity

According to Figure 7, the temperature changes have little effect on the temperature range and the storage capacity changes are relatively constant.

Adding TBAC to the system will increase the relative storage capacity (Figure 8).

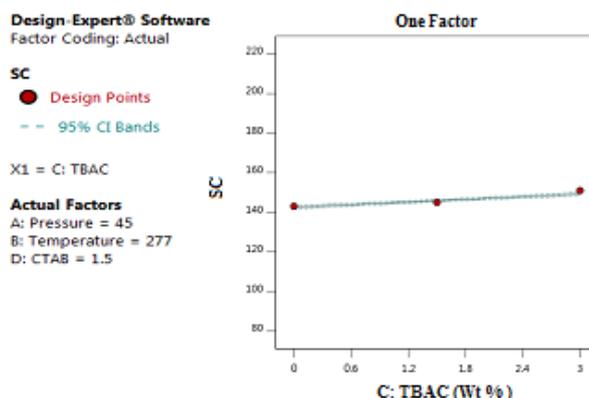


Figure 8. Effect of TBAC on storage capacity

Adding CTAB surfactant to the system results in increased storage capacity, which is slower than TBAC. And this is an effective story of this article.

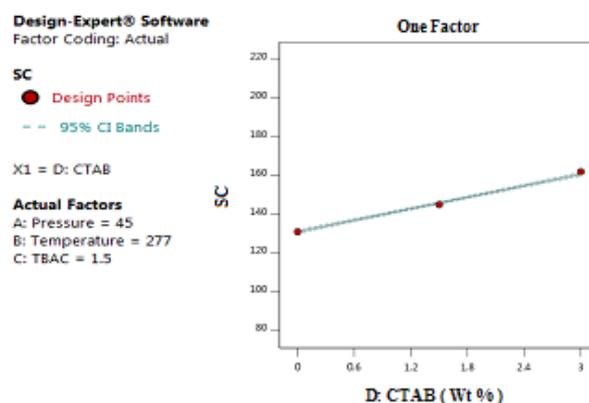


Figure 9. Effect of CTAB on storage capacity

## Conclusion

In this study, the effect of TBAC and CTAB surfactant on the induction time of hydrate formation and carbon dioxide storage capacity was investigated. Design Expert software was used to design the experiment. And finally, statistical analysis of the effective parameters on induction time of hydrate formation showed that TBAC can decrease the time of induction of hydrate format induction time of hydrate formation compared to other additives. Moreover, the most optimal reduction of induction time is related to the system containing the highest amount of additives. Also, regarding the effect of variables on the storage capacity of carbon dioxide gas, it can be concluded

that increasing the amount of CTAB surfactant and pressure, has the most impact on the increase of carbon dioxide storage capacity compared to the other parameters.

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