

Article

Efficient Storage of Methane in Hydrate Form Using Soybean Powder

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Abstract: Natural gas is a promising future source for the increasing energy demand. It is partially clean energy with fewer environmental impacts, and it is necessary to develop technologies to cater to the supply chain. Due to their inherent structural properties, gas hydrates or clathrate hydrates are promising materials for capturing and storing methane gas. In the present study, the experimental investigations were performed to assess the utilization of soybean powder (SBP) as a promoting additive compared to sodium dodecyl sulfate (SDS) for methane hydrate formation. The methane hydrate formation temperature and pressure with SBP are 277.8 ± 3.2 K, 7050.9 ± 76.2 kPa, similar to SDS 277.2 ± 0.3 K, 7446.3 ± 5.7 kPa in the non-stirred system. The gas uptake capacity is about 94.2 ± 4.5 v/v and 92.4 ± 4.6 v/v with SBP and SDS, which is ~60% of the practical, achievable limit. The time for the 90% of hydrate conversion is ~4.6 times higher for SBP than SDS. The more prolonged kinetics is ascribed to the complex constituents in the SBP. In contrast to the SDS solution, no foam was produced in the sample of the SBP solution. The current studies demonstrate that SBP can be utilized to develop cleaner and more effective promoters for methane hydrate formation without foam creation.



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1. Introduction

Due to its low levels of CO₂ emission, natural gas, primarily composed of methane, has seen increased demand in the previous several decades as a clean and safe energy source [1,2]. Modern, practical methane storage and transportation methods are required to use this priceless resource [3,4]. The natural gas hydrates are non-stoichiometric and resemble ice but vary in their characteristic properties. They are generally observed in the sediments at the ocean bottom and permafrost regions under the right thermodynamic circumstances. Lighter gases such as methane, ethane, and propane combine with water molecules to form hydrates. The cage network of the hydrate structure is developed by hydrogen bonding among the water molecules. Several polyhedral cages are connected to form three hydrate structures, namely structure I (sI), structure II (sII), and structure H (sH). The Van der Waals forces bind the gas molecules in the water cavities [5]. The gas hydrates offer a higher storage capacity (170.4 v/v) under milder conditions than liquefied natural gas (LNG) and compressed natural gas (CNG) [6,7]. They are considered potent materials for storing and transporting natural gas [3,8]. The working pressures are lower (3–5 MPa) for hydrates compared to the CNG (20–25 MPa). The hydrate-based gas storage (HBGS) is much safer than the LNG and CNG for transportation due to the self-preserving phenomenon of the hydrates in the metastable region [3,7,9].

The HBGS technology for gas storage and transportation is assessed at the laboratory scale. The real-time use of the technology is not yet in practice due to the stochastic nature of hydrate nucleation, lesser hydrate conversion, and prolonged process kinetics [10]. Hydrate

nucleation generally occurs at the gas–water interface due to sufficient contact between gas and water [11]. However, the hydrate coating separates the water phase from the gas phase, making it more challenging for gas molecules to enter the liquid phase. Consequently, the growth of hydrates and the process of turning water into hydrates will be delayed. Therefore, a crucial prerequisite for effective HBGS technology is quick hydrate formation in conjunction with high storage capacity. Several methods such as mechanical stirring, water spraying, and gas bubbling have been employed, and the use of various additives and thermodynamic and kinetic promoters have been assessed [12,13]. The mechanical methods hamper scaling it to industrial practice. The use of additives could affect the heat and mass transfer aspects, and the use of promoters (primarily thermodynamic promoters) could reduce the gas storage capacities [14]. Given that the formation of gas hydrate is an interfacial process, surfactants can improve mass transfer at the gas–liquid interface and remove the diffusion barrier between liquid and gas phases [11]. Amphiphilic chemicals known as surfactants can form a monolayer at the gas–liquid interface, arranging their hydrophilic groups in the liquid phase while stretching their hydrophobic groups into the gas phase. Several theories have been put out to explain the function of surfactants in enhancing gas hydrate formation, including micelle formation, adsorption, capillary, mass transfer enhancement, and water structure [15]. Sodium dodecyl sulfate (SDS) was a highly effective promoter for hydrate formation and development. SDS comprises a sulfate group as the polar head group and a lengthy alkyl chain as the hydrophobic component. The methane recovery is harmed by producing a lot of foam during the dissociation of methane hydrate in the presence of SDS [16]. Additionally, the widespread use of SDS will make environmental issues worse. Therefore, it is imperative to create bio-based promoters that aim to lessen foam creation during the formation and dissociation of gas hydrates. Recently, other substances have been researched as green promoters, including cyclodextrins, amino acids, lignin, potato starch, Arabic and guar gums, antifreeze, Chinese medicines, cros dextrin, sunflower oil, tomato juice, and carboxylate surfactants [17–20]. The present study aims to develop and assess novel bio-additives. The use of soybean powder is investigated, which is eco-friendly and could be an alternative to the typical surfactants, which have low biodegradability and foam generation in the hydrate formation. They have a lot of amino acids and are a great source of proteins [21]. This is the driving force behind the decision to use these powders. The edible seed of the annual legume soybean (*Glycine max*), often known as soja bean or soya bean, belongs to the Fabaceae family of plants. The chemical makeup of soybeans has relatively high concentrations of protein, conjugated carbohydrates, soybean oil, fatty acids, amino acids, and inorganic substances (minerals). Protein and fatty acid levels comprise 40% and 20% of these soybean constituents [22]. These soybean powders were compared to SDS to understand the promoting effect and the process of methane hydrate formation.

2. Experimental Method

2.1. Materials Needed for the Formation of Hydrate

The SDS powder was purchased from M/S Sigma Aldrich and utilized as received. The soybean seeds were purchased from the local market. The study employed methane gas purchased from M/S Bhuruka Gas Company, Bengaluru, India, that was 99.99% pure. In the sample preparation, deionized millipore water type 1 was utilized.

2.2. The Equipment and Steps Used to Form the Hydrate

A batch-style 100 mL high-pressure reactor (Amar Equipment Pvt. Ltd, Mumbai, Maharashtra, India) was used for the research. The isochoric technique was used to produce hydrates. In Figure 1, the experimental setup is shown. Before the sample was placed into the high-pressure reactor, it was properly cleaned. To prevent any contamination, great care was taken. The reactor chamber was loaded with the 29 mL sample. The soybean seeds were powdered using a mixed grinder. Deionized water and 0.5 wt% of the SBP and SDS were used to prepare the sample solution. The reactor head was then

placed and securely sealed with the help of the screws. Using an ISCO syringe pump, the experimental gas methane was pressured into the reactor through the inlet valve. The chamber was repeatedly pressurized (0.2 MPa) with the test gas prior to pressurizing the reactor. The reactor temperature can be raised or lowered using the chiller. As a coolant, a 2:1 mixture of water and glycol was utilized. An abrupt decrease in pressure and a temperature rise caused an exothermic reaction to signal the presence of hydrates. A platinum resistance thermometer was used to gauge the temperature within the vessel (Pt100). A WIKA pressure transducer (WIKA, type A-10 for pressure range 0–16 MPa) was used to measure the pressure. The temperature and pressure measurements had respective uncertainties of ± 0.5 K and 2%. At predetermined intervals, the pressure and temperature values were recorded.

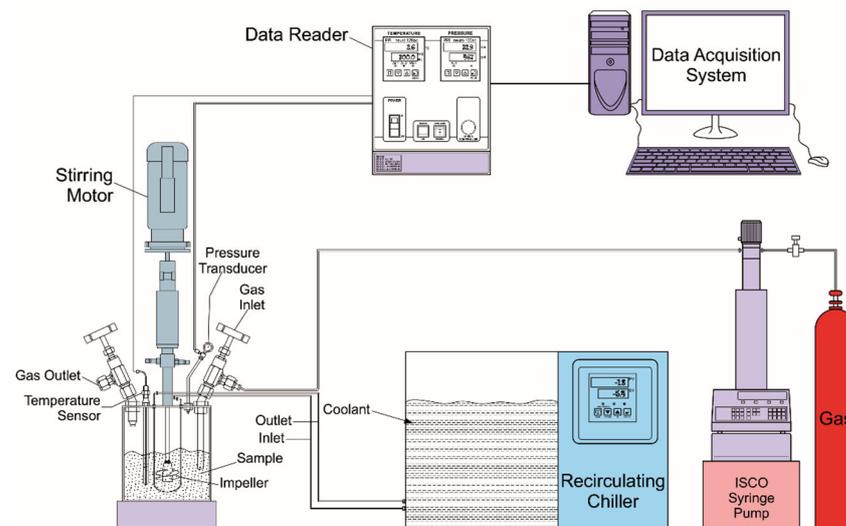


Figure 1. The conceptual, experimental plan used to synthesize methane hydrates.

2.3. Equations Used for Calculating and Inferring the Data

2.3.1. The Formula for Estimating How Much Gas Is Consumed during Hydrate Formation

The following equation describes the amount of gas (moles) present in the hydrate phase at time t during the experiment:

$$\Delta n_{H,t} = n_{g,0} - n_{g,t} = \left[\frac{P_0 V}{Z_0 R T_0} \right] - \left[\frac{P_t V}{Z_t R T_t} \right] \quad (1)$$

The compressibility factor Z is calculated using the Peng–Robinson equation of state. P —pressure; V —volume; T —temperature; R —gas constant. 0 —initial point; t —a point at any given time.

Throughout the trials, a constant gas volume (V) was assumed. Neglect is shown in the volume variations brought on by hydrate formation. The numbers $n_{g,0}$, and $n_{g,t}$ represent the quantity of feed gas in the gas phase and at the hydrate nucleation point, respectively.

2.3.2. The Equation for Calculating the Gas Uptake (v/v)

The volumetric gas uptake (v/v) was calculated using the following equation. It is defined as the “volume of gas/volume of water (hydrate + unreacted)”.

$$v_t = \frac{\Delta n_t \times 22,400}{(n_{H_2O} - 6.8 \Delta n_t) (MW_{H_2O} / \rho_{H_2O}) + (\Delta n_t \times MW_{hydrate} / \rho_{hydrate})} \quad (2)$$

where Δn_t is the mole of gas consumed, n_{H_2O} is the mole of water used, MW_{H_2O} is the molecular weight of water, ρ_{H_2O} is the density of water, and $MW_{hydrate}$, $\rho_{hydrate}$ are the CH_4 hydrate molecular weight and density calculated using the CSMGem application [5].

3. Results and Discussion

3.1. Methane Hydrate Formation

Scientists suggested a variety of chemicals to overcome the difficulties with hydrate formation. Thermodynamic boosters are substances that shift the phase equilibrium curve of gas hydrate toward lower pressures or higher temperatures. Meanwhile, kinetic promoters address the issues with gas hydrates' slow formation rate and long induction times. By lowering the gas–liquid surface tension or speeding up the solution's mass and heat transfer rates, kinetic promoters directly impact the amounts of gas consumed, rate of gas uptake, induction time, and storage capacity of hydrates. The need for new kinetic promoters is growing, and because green promoters are inexpensive, biodegradable, environmentally friendly, and non-toxic, researchers have taken an interest in them. The experiments were carried out in a non-stirred isochoric reactor and are shown in Figure 1. The impeller was turned off in all experiments. The initial operating conditions were at 300 K and 7.8 MPa.

The first step involves mixing 0.5 wt% of the SBP with water to prepare the sample. The SDS sample was prepared comparably. Figure 2 shows the pressure–temperature tracks of the formation and dissociation of methane hydrate synthesized with the SBP (a) and SDS (b). The blue and red dots show the formation and dissociation of hydrates, respectively. The black line represents the phase equilibrium curve of the sI CH₄ hydrate produced by the CSM GEM application [5]. Interestingly, the average hydrate nucleation with the SBP occurs at about 277.8 ± 3.2 K, near the SDS hydrate nucleation point of 277.2 ± 0.3 K. An abrupt drop in pressure and an exothermic heat release are signs of hydrate nucleation. These powders do not change the thermodynamic conditions of the hydrate system, as evidenced by the fact that the hydrate dissociation proceeds from the equilibrium of the sI methane phase.

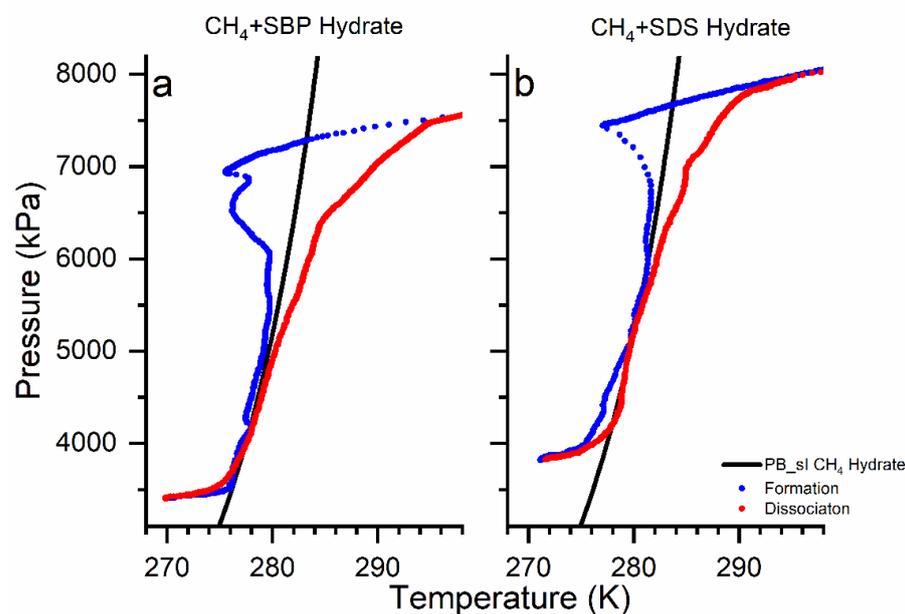


Figure 2. Pressure temperature trajectories of the methane hydrate formation (blue) and dissociation (red) in the presence of soybean powder (a) and sodium dodecyl sulfate (b).

The faster heating causes the dissociation at higher pressures to deviate from the line of phase equilibrium. It could be avoided if the dissociation is carried out at 0.1 K/H. The pressure–temperature trajectories for the SBP and SDS systems are similar. The amount of subcooling (subcooling is used to describe the difference between the phase equilibrium temperature and the experimental formation temperature under operating pressure) needed for forming hydrates with SBP and SDS is 5.1 ± 3.1 K and 6.4 ± 0.3 K, respectively. The experimental data for the methane hydrate system in the presence of SBP and SDS are

shown in Table 1. This demonstrates that, despite having many minerals, proteins, amino acids, and fatty acids as bio-components, SBP did not exhibit a significant thermodynamic divergence for the formation of hydrates in comparison to SDS [22].

Table 1. The table shows the methane hydrate formation conditions, uptake kinetics, subcooling, and hydrate yield in the presence of SBP and SDS systems.

Sample	T _{formation} (K)	P _{formation} (kPa)	Subcooling (K)	T ₉₀ Uptake Kinetics (min)	Hydrate Yield (v/v)
SBP	277.8 ± 3.2	7050.9 ± 76.2	5.1 ± 3.1	144 ± 4.2	94.2 ± 4.5
SDS	277.2 ± 0.3	7446.3 ± 5.7	6.4 ± 0.3	31 ± 5.2	92.4 ± 4.6

3.2. Methane Gas Uptake Capacity

Gas consumption demonstrates the hydrate's capacity to capture gas molecules in cages. The amount of gas consumed into the hydrate cavities at the beginning conditions ~7.8 MPa with the SBP and SDS is shown in Figure 3. The gas uptake capacity is an essential indicator of hydrate capability in gas storage applications. The ideal gas hydrate yield upon dissociation would be 160–180 times its volume in gas. Based on some model calculations for CH₄ hydrates, the ideal gas intake capacity is 170.4 v/v (ideal). The realistic, reachable limit is 157.5 v/v, although filling all the cages is difficult [5].

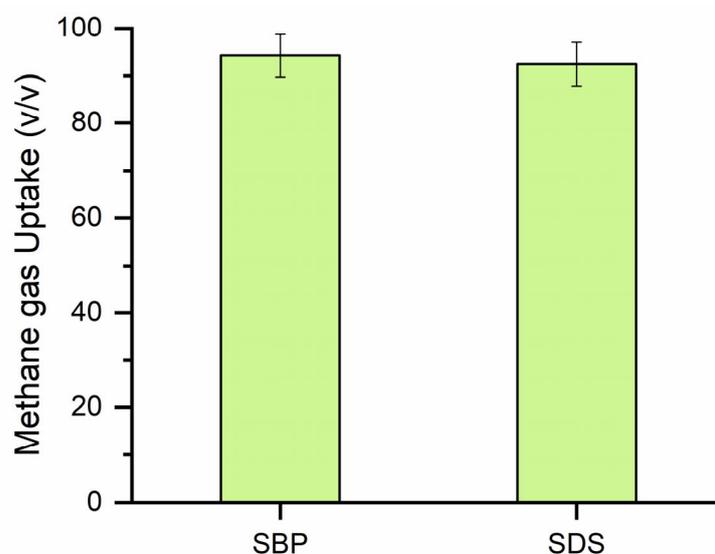


Figure 3. The figure shows the amount of methane gas uptake (v/v) in the presence of SBP and SDS samples.

With the chosen experimental configuration and the solution volume, the amount of methane uptake with the SBP and SDS is 94.2 ± 4.5 v/v and 92.4 ± 4.6 v/v. The total gas consumed with SBP and SDS is similar and corresponds to ~60% of the practical, achievable limit. The gas consumption and storage capacity were calculated using Equations (1) and (2). An organic compound, SDS, yields a similar hydrate conversion using a lesser quantity (0.5 wt%) of the SBP, a natural powder. The results are generally positive, and soybean powders would be a better option if they have quicker gas uptake kinetics and are not foaming.

3.3. Methane Gas Consumption Kinetics

The process kinetics or the gas uptake kinetics demonstrates the ability to adapt the hydrate process in real-time, much as the gas uptake capacity demonstrates the hydrate efficiency in storage applications. The process kinetics with the SBP and SDS for 500 min are shown in Figure 4. The green and brown lines correspond to the methane uptake

(mol/mol) with SBP and SDS samples. The line represents the average of three cycles, and the shaded portion is the standard deviation.

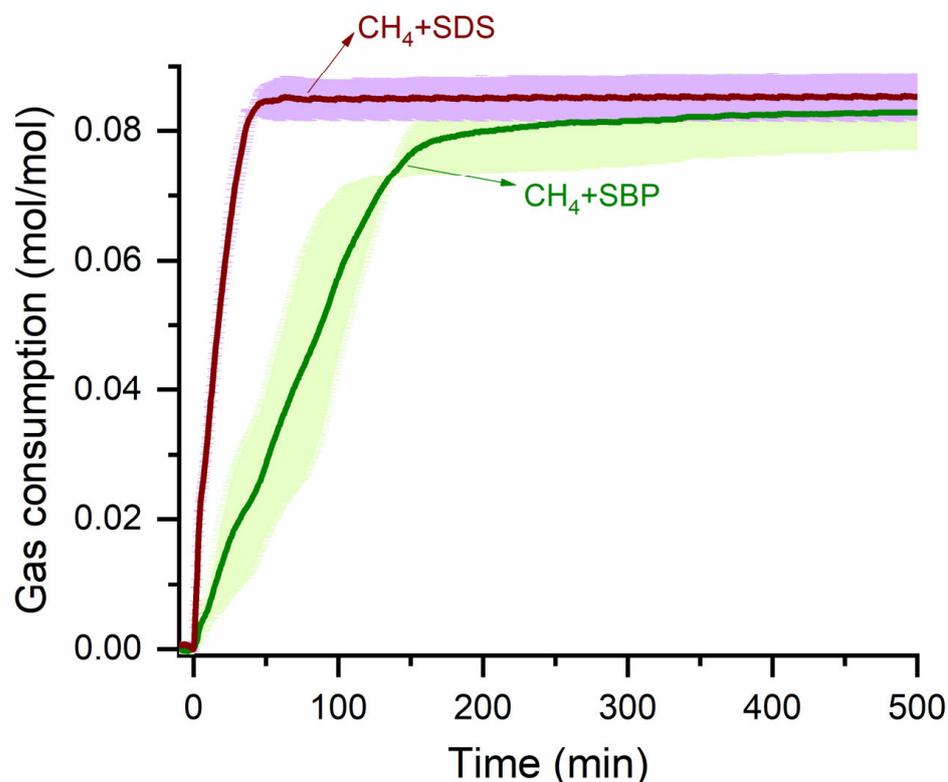


Figure 4. The figure shows the impact of SBP (green line) and SDS (brown line) on the amount of methane consumed per mole of feed water. The line is the average, and the shaded region is the standard deviation of the repeat cycles.

The results indicate that the amount of gas consumed in both the systems SBP and SDS are similar. The graph clearly shows that the time for 90% of the kinetics with the SDS system is 31 ± 5.2 min, whereas with the SBP is 144 ± 4.2 min. In SBP, compared to SDS, the hydrate growth kinetics is approximately 4.6 times delayed. The SBP is a constituent of many components, which include proteins, carbohydrates, and minerals [22]. The soybean, also known as glycine max, is a kind of legume that is widely farmed and has several purposes. The major constituents of soybeans are proteins. Among them, glycinin and β - and γ -conglycini are majorly available, and other proteins such as urease and cytochrome are lesser in quantity [21]. The composition of amino acids affects how biologically active proteins are since amino acids contain the information required to create proteins. The SBP contains 18 amino acids that are essential and non-essential for the human body. The amino acid composition in SBP is listed in Table 2 below [21]. It is seen that major amino acids are included in the SBP. The minor component is the cysteine and tryptophan, with 1.1 g/100 g of SBP and the higher composition is the aspartic acid and glutamic acid, with 10.2 and 16.4 g/100 g of SBP. Recent research has revealed that using mixed amino acids indicates a distinct behavior for the CH₄ and CO₂ gases [23]. The amino acid concentrations show synergistic and antagonistic behavior for hydrate formation depending on their critical concentration. In a mixed amino acid system, the hydrate inhibition is surpassed when a threshold quantity of the promoting amino acid concentration is increased and vice versa [23–25]. Similar to this phenomenon, it can be determined that glutamic acid, which is present in more significant concentrations than the other amino acids in the SBP system, may have a greater influence on the system's function. For methane hydrates, Liu et al. (2015) [26] conducted studies with various amino acids at lower concentrations (0.5 wt%). The methane uptake with glutamic acid (0.5 wt%) was

~60% of the optimum uptake capacity (151 mg/g), and the uptake kinetics were somewhat slower than those of the solid former amino acids methionine and phenylalanine. The SBP system's slower uptake kinetics characteristics more closely reflect those of the glutamic acid system, indicating that it has an impact on hydrate formation. The other amino acid components will also have an impact on the hydrate system, although glutamic acid may have a greater impact due to its larger concentration.

Table 2. The table shows the amino acid components in the soybean powders. The data is taken from Garcia, Reprinted/adapted from Ref M.C. et al. 1997 [21].

S.No	Amino Acid	Content (g/100 g Protein)
1	Cysteine	1.1
2	Tryptophan	1.1
3	Methionine	1.2
4	Histidine	2.3
5	Threonine	3.3
6	Tyrosine	3.3
7	Glycine	3.7
8	Alanine	3.8
9	Isoleucine	4.3
10	Valine	4.4
11	Proline	4.5
12	Phenylalanine	4.6
13	Serine	4.6
14	Lysine	5.5
15	Arginine	6.7
16	Leucine	7.2
17	Aspartic acid	10.2
18	Glutamic acid	16.4

3.4. Reusability and Foaming of Sample Solution

Surfactants such as sodium dodecyl sulfate are frequently utilized to hasten the formation of hydrates. These aid in enhancing the kinetics of hydrate formation, and the induction time is considerably shorter than with pure hydrates [15]. Reusing the hydrate-forming solutions is challenging in systems containing SDS because of significant foaming, but not in systems containing SBP. Figure 5 shows the photographs of the sample solution taken after the hydrate dissociation. The SBP sample is clear and can be reused for multiple cycles. In contrast, the SDS solution creates a lot of foam and is highly difficult to reuse. Surfactant foaming at high levels impedes gas release from hydrates [16]. The hydrates crystallized into a solid phase with SBP and SDS samples. The soybean powder would be a preferable option for the methane hydrate process because of its non-foaming and yielding higher hydration conversion. Additionally, hydrate nucleation has a lower stochastic nature and most likely has modest uptake kinetics.

The appearance of sample solution after methane hydrate dissociation.



Figure 5. The figure shows the photographs of SBP and SDS solutions after the methane hydrate dissociation. The SDS system is where foaming is most frequently seen.

4. Conclusions

The results of the current study were compared to those obtained using sodium dodecyl sulfate as they evaluate the utilization of soybean powder for the promotion of methane hydrate formation. The formation temperatures for the SBP and SDS systems were 277.8 ± 3.2 K and 277.2 ± 0.3 K, respectively. The gas uptake capacity was about 94.2 ± 4.5 v/v and 92.4 ± 4.6 v/v with SBP and SDS, which is ~60% of the practical, achievable limit. The time for the 90% of hydrate conversion was ~4.6 times higher for SBP than SDS. Under the conditions examined here, these natural materials can hold up to about 94.2 v/v STP of methane gas. The gas uptake kinetics are modest, and the retardation is ascribed due to the various constituents present in the SBP. Remarkably these SBPs are non-foaming, biodegradable, renewable, and inexpensive. However, there is currently no clear data on the economics of using bio-powders for gas storage and transportation. Using bio-powders is a novel method for storing a gas that has the benefit of utilizing solely natural, regenerative resources. There is much potential to discover the appropriate bio and renewable promoters, such as soybean powders, to form methane hydrates with a 100% conversion, opening the door for the real world for methane or natural gas storage and transportation in a hydrate form.

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