Journal of Materials Science and Chemical Engineering, 2026, 14(5), 1-12
https://www.scirp.org/journal/msce
ISSN Online: 2327-6053
ISSN Print: 2327-6045

Coatings for Controlled Formation and Adhesion of Gas Hydrates in Pipelines

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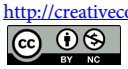
How to cite this paper: Diakite, D.A. and Sidibe, M.S. (2026) Coatings for Controlled Formation and Adhesion of Gas Hydrates in Pipelines. *Journal of Materials Science and Chemical Engineering*, **14**, 1-12.
https://doi.org/10.4236/msce.2026.145001

Received: March 30, 2026
Accepted: May 19, 2026
Published: May 22, 2026



Abstract

The formation of gas hydrates (GHs) in pipelines remains a major challenge to flow assurance in the oil and gas industry due to the risk of blockages during production and transportation. Despite their energy and environmental potential, gas hydrates can form and clump together, causing blockages and operational disruptions. These blockages can occur at various stages of hydrocarbon handling, including production, processing, and transportation, and also pose safety risks in pipelines and subsea production systems. Controlled hydrate formation, based on the management of thermodynamic and kinetic factors, is desirable. It allows the formation and development of hydrates to be managed in a controlled manner to prevent or reduce blockages within pipelines in order to ensure safe transport of suspensions, rather than seeking to eliminate them completely. Managing GHs through controlled formation methods improves the efficiency of oil and gas transportation in pipelines and reduces the risk of incidents due to blockages and shutdowns. In this context, environmentally friendly surface coatings appear as promising alternatives to current methods that use chemical additives and active heating, which are often costly and environmentally harmful. This review critically examines the progress made over the last decade. This study highlights the key role of wettability, roughness and interfacial surface properties in regulating the adhesion behavior of hydrates.

Keywords

Gas Hydrate, Pipeline, Hydrate Adhesion, Wettability, Coating

1. Introduction

GHs are ice-like crystalline substances composed of gas molecules such as CH_4 and CO_2, trapped in cavities due to hydrogen bonding in water and Van der Waals forces at low temperatures and high pressures. They are found in permafrost and continental margins, containing quantities of organic carbon that can exceed those of all traditional fossil fuels, highlighting their potential as a low-CO_2 energy source [1]. Although GHs pose a challenge to the oil and gas industry, their ability to form and dissociate easily, while storing large quantities of gas, offers opportunities for a variety of applications, including gas capture and storage, transportation, separation, refrigeration and seawater desalination, the production of carbonated solid foods, and energy production [2]. The structure of GHs varies according to the size of the guest molecules (**Figure 1**): methane (CH_4) and carbon dioxide (CO_2) form cubic I (sI) hydrates, butane (C_4H_{10}) forms cubic II (sII) hydrates, and hexagonal H (sH) hydrates result from a mixture of hydrocarbons such as CH_4 and cyclopentane [3] [4]. Each structure is characterized by a set of various cavities that accommodate the guest molecules. Among these, we find specific configurations such as the pentagonal dodecahedron (5^{12}), the tetrakaidecahedron ($5^{1,2}6^2$), the hexakaidecahedron ($5^{1,2}6^4$), the irregular dodecahedron ($4^35^66^3$) and the icosahedron ($5^{1,2}6^8$), as shown in **Figure 2**. These cavity variations play a crucial role in the physical and chemical properties of clathrate hydrates, influencing their application potential in various scientific and industrial fields.

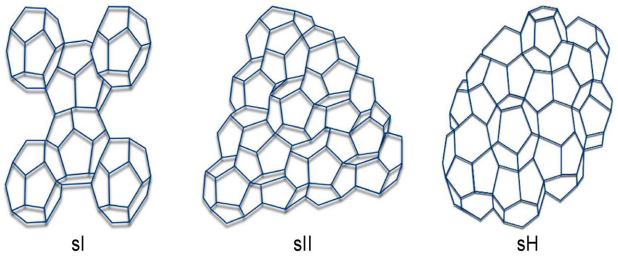


Figure 1. Structures of clathrate hydrates: Structure I (sI), Structure II (sII) and Structure H (sH) [2].

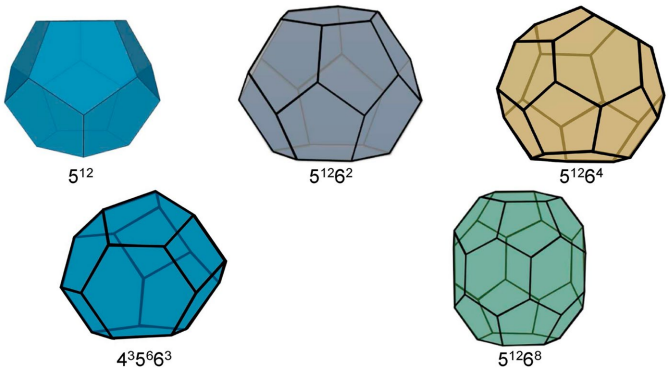


Figure 2. Clathrate hydrate cavities: pentagonal dodecahedron (5^{12}), tetrakaidecahedron ($5^{1,2}6^2$), hexakaidecahedron ($5^{1,2}6^4$), irregular dodecahedron ($4^35^66^3$) and icosahedron ($5^{1,2}6^8$) [2].

During deepwater oil and gas exploration, high-pressure, low-temperature conditions are common. Water present in pipelines promotes hydrate formation, which can lead to blockages. To prevent HGs formation during production, industries use costly technologies, including dewatering and the injection of chemical inhibitors [5]. While the latter is considered the most cost-effective method, its use at high doses can harm aquatic ecosystems. However, recent regulatory strategies for the search for low-dose inhibitors remain insufficient in terms of economic, energy, and environmental efficiency. Therefore, a recent approach involves applying a hydrophobic treatment to surfaces that minimizes hydrate adhesion and facilitates the transport of slurries from hydrate stability zones without negatively impacting production processes. In this regard, modifying the hydrophobic properties of surfaces appears to be a promising approach to minimizing the risk of hydrate formation. By providing moisture resistance, the hydrophobic coating reduces the likelihood of hydrate formation and ensures stable operation of pipeline systems under conditions where there is a risk of formation. However, creating surfaces that allow for reliable hydrate management remains a technical challenge.

This review focuses on recent innovations in gas hydrate management, particularly in the oil industry. It highlights the importance of hydrophobic coatings and their impact on environmental sustainability. Furthermore, it explores new applications, notably in sludge dewatering and the control of hydrate nucleation and growth, emphasizing technological advancements in these areas.

2. Deposition and Adhesion of Gas Hydrates to Pipeline Walls

The formation and deposition of hydrates in pipelines can lead to blockages, threatening the safety of oil and gas production and transportation [6]. The conceptual model of hydrate blockage in oil pipeline systems indicates that the agglomeration of liquid-phase hydrates on pipe walls is the primary cause [7]. However, recent research has shown that, in addition to agglomeration, the growth of hydrate deposits on the walls also contributes to increased pressure in the pipeline during blockage. Hydrate deposits form through the deposition and adhesion of hydrate particles or aggregates to surfaces. Nevertheless, these deposits can affect various systems, acting as blockages in pipelines or influencing fluid properties in transportation systems. Therefore, directly measuring the adhesion strength between hydrates and solid surfaces is essential for understanding hydrate deposition mechanisms, thus providing important information on their interaction and behavior in various industrial and environmental contexts [8]. Adhesion strength is strongly influenced by two main factors: surface roughness and the nature of the materials [9]. Indeed, surfaces characterized by low roughness and high hydrophobicity exhibit weaker adhesion strength. Preventive measures against hydrate adhesion are of great interest for slowing down or minimizing undesirable hydrate formation.

Hydrate adhesion is the most important step in the blocking process. To effec-

tively prevent hydrate blockage, even under conditions of intensive hydrate formation, it is recommended to use methods aimed at reducing the adhesion of hydrated formations. In predominantly aqueous systems, a thin film of water forms on the surface and acts as a lubricant and barrier, significantly weakening the adhesion of hydrates and their deposition on pipeline walls [8] [10] [11]. Despite the recognized effectiveness of hydrophobic solid surfaces for this purpose, the underlying molecular mechanism of promotion remains poorly understood [12]. To understand the atomic mechanisms of adhesion between hydrates and solid surfaces, Ma *et al.* [13] used molecular simulations that revealed that hydrate adhesion to solid surfaces is influenced by the structure of the intermediate layer, maintained by growth competition, and regulated by the concentration of host molecules. The results indicate that the surface density of the water network and the adsorption of host molecules are key factors in adhesion strength. Analysis of adhesion forces shows that the adhesion strength of ice is approximately five times stronger than that of hydrates, highlighting significant differences between water structures such as ice and hydrates. Chenru *et al.* [14] developed an innovative method to quantitatively measure the adhesion of methane hydrates to an oily substrate, using a high-pressure microforce device. The results indicate that the adhesion force increases rapidly at the beginning of hydrate deposition with formation time, reaching maximum values of 887.26 ± 78.62 kPa, 1013.12 ± 98.4 kPa and 1480.25 ± 128.62 kPa at respective subcoolings of 1˚C, 4˚C and 7˚C. Chenwei *et al.* [15] used a custom-designed apparatus to measure the adhesion strength of sintered cyclopentane (CyC5) hydrate deposits in the presence of thermodynamic inhibitors and at low doses. The results indicate a decrease in adhesion strength from 69.82% to 97.06% with ethylene glycol and from 40.24% to 94.36% with glycerol, as the concentration increases from 2% to 6%. For DBSA and Span 80, adhesion strength increases at concentrations below 0.01%, then drops drastically above this level. Modified models have been proposed to better predict hydrate adhesion, taking into account crystal morphology and the hydrate formation rate.

Exploration results from hydrate deposits show that within the stability zone of natural gas hydrates, different phases coexist: solid hydrates, sediments, aqueous solutions, and free or dissolved gas [16]. Therefore, the formation and dissociation of hydrates take place in complex multiphase systems, influenced by interfacial interactions. The interfacial properties and mechanisms of gas hydrates are fundamental for controlling particle-surface interactions, influencing the energy applications of clathrate hydrates. Preventing interparticle agglomeration and particle deposition is essential to avoid blockages during hydrocarbon exploration and transport in subsea pipelines, processes determined by cohesive forces between particles and adhesion to surfaces [17]. Zhang *et al.* [18] developed a macroscopic mathematical model to predict the nucleation kinetics of gas hydrates, taking into account the interfacial properties of sediments. He distinguishes two formation scenarios: in hydrophilic sediments, nucleation begins at the wall and progresses toward the pore center, while in hydrophobic sediments, it starts at the center and

grows toward the walls, slowly forming a hydrate phase at the intersections of the gaseous, liquid, and solid phases. Mathews *et al.* [19] used molecular dynamics to evaluate the surface stresses related to the interfacial tension of methane/ethane sII hydrates, as well as gas mixtures, at various temperatures and pressures. It was observed that surface tension increases with temperature, balancing the interactions between water, solid, and gas. The molecular dipole highlights the complex behavior of water molecules in a thin, compressed pre-melting layer, which acts as a quasi-liquid.

Anti-caking agents (AAs), which are low molecular weight surfactants, are one strategy for controlling or preventing the formation of hydrate plugs. They work by inhibiting the agglomeration of small hydrate particles, thus preventing the formation of large plugs. Interparticle cohesive forces within hydrates play an important role in studying their interfacial characteristics and their tendency to agglomerate. Hu and Koh [20] developed a modified high-pressure micromechanical force measurement (HP-MMF) device to measure the cohesive force of hydrates at high pressure and low temperature in deepwater pipelines. The hydrate cohesive force decreased significantly from a baseline value of 23.5 ± 2.5 $mN*m^{-1}$ to less than 0.05 $mN*m^{-1}$ when the concentration of a high-performance amino acid (AA1) increased from 0.25% to 2%. The cohesive force measurements simulated shutdown scenarios, indicating that prolonged contact times can lead to higher cohesive forces and potential system failure due to irreversible interparticle interactions in the event of underdosing. Furthermore, the underdosing scenarios identified for AA1 in model liquid hydrocarbons showed that salt ions improve the performance of AA1, although they only slightly reduce the cohesive strength with AA2 at the same level of undercooling. Similarly, in 2021, Hu *et al.* [21] used a high-pressure microforce apparatus to investigate the influence of the molecular structure of hydrate anti-caking inhibitors (AAs) on gas hydrate crystal interactions. Four AA molecules, including quaternary ammonium salts with long (R1) and short (R2) chains, were tested in the presence of n-dodecane and n-heptane. All AAs reduced interparticle cohesive forces (baseline value: 23.5 ± 2.5 $mN*m^{-1}$), with AA-C12 exhibiting the best performance. The results showed that AAs with longer R1 alkyl chains create denser barriers and achieve a higher packing density when their chain length aligns with that of the liquid hydrocarbons on the surface of the gas hydrates. Increased salinity enhances the effectiveness of AAs and mitigates the impact of prolonged contact time, which otherwise increases cohesive forces.

3. Hydrophobic Surface Treatments and Coatings

In the oil and gas industry, various methods for managing hydrate risks have been implemented, including operational and chemical approaches. Thermodynamic conditions (temperature and pressure) are controlled to prevent hydrate formation using techniques such as depressurization, heating, and dehydration. The injection of chemical inhibitors, such as thermodynamic and kinetic inhibitors,

and anti-caking agents, is also employed. However, these methods have economic and environmental limitations, hence the need to develop more environmentally friendly and efficient solutions. Consequently, various coatings and surface treatments can play a significant role in modifying the adhesion of hydrates to solid surfaces. These methods influence how hydrates interact with materials, potentially enhancing or reducing their adhesion.

3.1. Hydrophobic Functionalized Surfaces

The systematic functionalization of solid surfaces by modulating their hydrophobicity is an effective method against clathrate hydrate formation and oil pipeline clogging [22]. The application of functionalized coatings can reduce the adhesion of hydrates to surfaces, thus allowing hydrodynamic shear stresses to detach deposits and prevent clogging. Zhang *et al.* [23] investigated the use of functionalized nanoparticles as an alternative to anti-caking agents to create a hydrate suspension. These nanoparticles, particularly 256 nm silica spheres (**Figure 3**), are considered environmentally friendly and recyclable. The results reveal that nanoparticles with moderate hydrophobicity stabilize emulsions and influence the morphology of cyclopentane hydrates, preventing their agglomeration. Furthermore, they slow viscosity while improving water conversion and maintaining low viscosity under steady-state conditions. The study highlights the significant potential of nanoparticles to prevent the agglomeration of gas hydrates under real pipeline conditions.

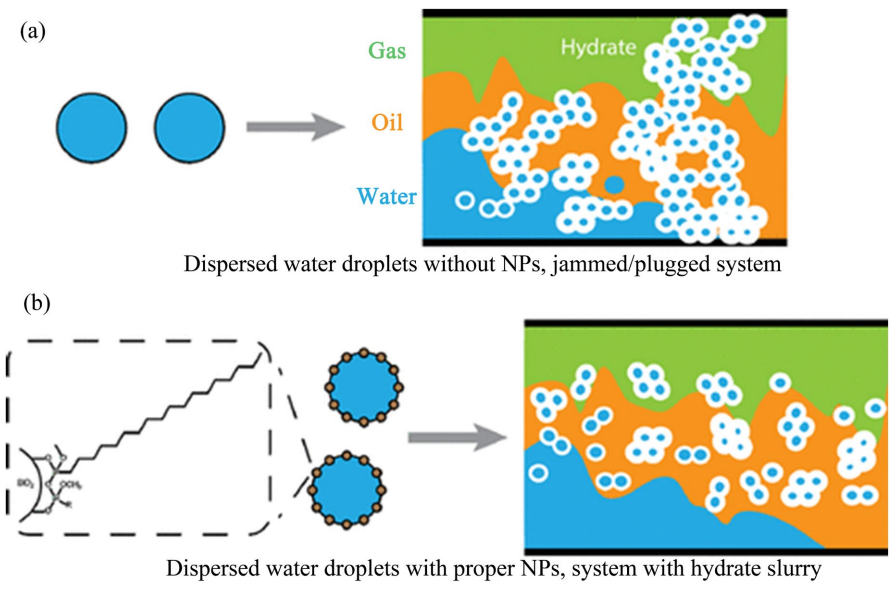


Figure 3. (a) Dispersed water droplets without nanoparticles, jammed/clogged system; (b) Dispersed water droplets with corresponding nanoparticles, system with hydrate suspension [23].

In the petroleum industry, a superamphiphobic coating with anti-adhesive properties could be a promising solution to paraffin deposition and flow obstruc-

tion problems, ensuring reliable transport in pipelines. However, the main challenge in achieving this remains reconciling the coating's non-wettability with its durability. Peng *et al.* [24] fabricated a multifunctional superamphiphobic coating using a simple sputtering method, incorporating highly fluorinated palygorskite@SiO_2 (Pal@SiO_2-F) composite fillers obtained by in-situ growth of SiO_2 on the surface of Pal and by chemical modification. These fillers are then inserted into polyethersulfone (PES) and poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) to create micro/nano-hierarchical structures with papillae and networks. The coating formed a stable air film that served as a non-stick and thermal barrier, with excellent adhesion in petroleum systems. It maintained its hydrophobicity after 2000 cycles and 30 days in crude oil, effectively in pipelines, precipitating wax at 90.9% and remaining resistant to mechanical damage and corrosion. Chen *et al.* [25] used the sol-gel process to obtain multifunctional superamphiphobic composite coatings. By modifying the tetraethyl orthosilicate (TEOS) content in an alkaline medium, controlled crosslinking between 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDTES) and polydopamine-functionalized graphene oxide nanosheets (PDA-GO) was achieved, producing robust nanocomposite powders. Formulated with lamellar graphite and a fluorocarbon resin, these powders yielded coatings with superior hydrophobicity. The coatings exhibited a static contact angle of 153.33˚ ± 0.74˚ and a rolling angle of 5.79˚ ± 2.02˚ for oil droplets, maintaining their performance after 67 days of immersion in acidic, alkaline, and saline solutions. The integrated two-dimensional graphene oxide also improves electrical resistivity, thermal response, and photothermal conversion capabilities. Zhang *et al.* [26] developed a spray-applied, multifunctional amphiphobic coating, PF/ZSM-5, from ZSM-5 zeolite modified with 1h,1h,2h,2h-perfluorooctyltriethoxysilane and a polyethersulfone adhesive. The coating effectively repels water, oil, paraffin, N-hexadecane, and petroleum, inhibits hydrate nucleation, and reduces hydrate adhesion to 0 mN/m. The coating is also resistant to fouling and corrosion, maintaining low hydrate adhesion even after 20 and 300 days in crude oil and tetrabutylammonium bromide (TBAB) solutions, respectively. Durability is attributed to the unique architecture and amphiphobic properties that form stable air cushions at the solid-liquid interface.

3.2. Superhydrophobic Coatings

Recently, the development of superhydrophobic coatings for pipelines has been recognized as effective in reducing gas hydrate fouling. However, these surfaces promote gas enrichment and hydrate growth, while also being brittle and susceptible to damage, thus compromising their durability. The adoption of hydrophobic surfaces requires robust coatings for extreme environments. Zhang *et al.* [27] presented a biomimetic CeO_2/pDA@X80 coating, obtained by static self-assembly, characterized by exceptional superhydrophobicity with contact angles of 154.7˚ and 155.5˚ in air and oil, respectively, and slip angles of 3˚ and 2˚. It demonstrates mechanical stability after a 2-hour abrasion test and meets adhesion class 2. Mi-

cromechanical tests show that the adhesion strength of hydrates to the coating is approximately 98.9% lower than that of X80, which minimizes hydrate adhesion after abrasion. Unlike X80, where water adheres strongly, CeO_2/pDA@X80 allows for easy water detachment due to its superhydrophobic surface. In this article, Dong *et al.* [28] presented the creation of a biomimetic superhydrophobic coating on an X90 steel substrate via copper electrodeposition, hydrothermal treatment to generate a copper oxide layer, and modification with stearic acid. The coating exhibits static contact angles of 160˚ ± 3.1˚ for water and 170.7˚ ± 2.5˚ for cyclopentane, demonstrating its water-repellent properties. Analysis of the cyclopentane hydrate morphology shows that those formed on the coating have a spherical morphology, reducing the contact area and the adhesion strength to the solid surface, potentially due to this decrease in contact area.

Sojoudi *et al.* [29] proposed bilayer coatings of polydivinylbenzene (pDVB) and polyperfluorodecylacrylate (pPFDA) applied by initiated chemical vapor deposition (iCVD) to reduce the adhesion of ice and hydrates to silicon and steel. These polymer films, with a contact angle greater than 150˚ and a low hysteresis of 4˚, exhibit RMS roughness of 178.0 ± 17.5 nm on silicon and 312.7 ± 23.5 nm on steel. The coatings reduce the adhesion strength of ice from 1010 ± 95 kPa to 180 ± 85 kPa and that of cyclopentane hydrate from 220 ± 45 kPa to 34 ± 12 kPa. Durability is confirmed by erosion tests and adhesion/disadhesion cycles. Yin *et al.* [30] fabricated a robust, superhydrophobic, 3D porous skeleton inspired by glass sponges that resolves the conflict between hydrate nucleation inhibition and superhydrophobicity. Its high specific surface area allows for an increased content of terminal hydroxyl groups, inhibiting the formation of new hydrates without compromising superhydrophobicity. Molecular dynamics simulations show that these groups disrupt the arrangement of water molecules, prolonging hydrate induction time by 84.4% and reducing adhesion strength by 98.7%. Furthermore, it retains its inhibitory and anti-adhesion properties even after 4 h of erosion at 1500 rpm, opening up potential applications in the oil and gas industry, as well as in carbon capture and storage.

3.3. Polymer Coatings

With technological advancements, the use of pipeline coatings is being promoted for preventing hydrate formation in oil and gas pipelines. Su *et al.* [31] conducted adhesion tests on various materials, revealing that the PTFE/PPS composite coating reduces the adhesion of hydrate particles at low temperatures. As subcooling increases, the adhesion strength rises from 8.36 $mN \cdot m^{-1}$ to 10.26 $mN \cdot m^{-1}$, but remains on average 68% lower than that of an uncoated substrate. E-51 epoxy resin and polyurea coatings exhibit good properties, but are less effective than PTFE/PPS.

To reduce the adhesion of hydrates to pipeline walls, Fan *et al.* [32] used hexagonal boron nitride (HBN), polydimethylsiloxane (PDMS), and a hydrophobic silicon dioxide (SiO_2) fluorinated coating as coating materials. The coatings were

tested on four substrates: X70 steel, X80 steel, zirconia (ZrO_2) plate, and tin-plated steel sheet. A micromechanical force measuring (MMF) device was used to measure the adhesion strength of tetrahydrofuran (THF) hydrate particles to the substrates. The results indicated that the fluorinated coating on the X70 substrate reduced the adhesion strength of THF from 1% (0.0152 N/m) to 4% (0.0030 N/m) of SiO_2, while the adhesion strengths were 0.0105, 0.0027, and 0.0043 N/m for X80 (bare), X80 + HBN, and X80 + PDMS, respectively. The PDMS + SiO_2 (1 - 4%) coating slowed hydrate growth. Further study in a high-pressure reactor showed that the PDMS + SiO_2 (4%) coating on a tinplate substrate inhibits methane hydrate growth, unlike the complete coverage observed on an uncoated layer. This coating also limits the binding of THF and methane hydrates, with surface morphology being a factor influencing hydrate particle adhesion and growth.

Xu *et al.* [33] measured the adhesion strength of a semi-clathrate hydrate (tetrabutylammonium bromide hydrate) on four solid surfaces: E235B carbon steel, E355CC low-alloy steel, SUS304 stainless steel, and polytetrafluoroethylene. The results show that the adhesion strength is inversely proportional to the wettability of the surfaces, indicating that hydrophobic materials decrease the adhesion of the hydrate. In contrast, surface roughness influences the adhesion of hydrates differently on hydrophilic and hydrophobic surfaces, with a 123.6% increase in adhesion on the hydrophilic surface of SUS304 stainless steel versus only 21.5% on the hydrophobic surface of polytetrafluoroethylene when the roughness increases from 3.2 μm to 12.5 μm. Wang *et al.* [34] designed a hydrophilic, low-adhesion surface inspired by the skin of an onion bulb. This surface significantly reduces the adhesion of cyclopentane hydrate (CyC5) from 95 kPa to 4.7 kPa. Furthermore, it maintains a low adhesion of 8.7 kPa after 20 hydration/dehydration cycles, and its effectiveness is enhanced by the addition of a regenerable artificial cuticle layer, achieving an adhesion of only 2.9 kPa. Tang *et al.* [35] presented a hydrophilic gel coating, Zr^{4+}/DMSO-Gel, that delays the nucleation and growth of methane hydrates on solid surfaces. Microscopic analyses showed partial decomposition of the hydrates, creating a thick water layer that reduces adhesion. Raman spectroscopy revealed that the coating promotes hydrogen bond interactions with water molecules, increasing bound water (BW) at the surface and reducing tetra-coordinated hydrogen-bonded water (4-HBW), which inhibits hydrate formation. These results pave the way for anti-hydrate coatings and new strategies to prevent gas pipeline fouling.

4. Conclusions

The formation and accumulation of clathrate hydrates can cause significant blockages in oil and gas production and transportation systems. These blockages pose major challenges for offshore oil platforms during operation, impacting the efficiency and safety of operations in these complex subsea and Arctic environments. Traditionally, preventing hydrate buildup on pipeline walls has relied on the use of chemicals and thermal methods. While these techniques are effective, they have

significant drawbacks, including high costs and accelerated environmental degradation.

This review highlights surface engineering approaches, particularly hydrophobic and amphiphobic coatings, which represent promising solutions for reducing hydrate adhesion. Unlike methods that aim to completely eliminate hydrate formation, these techniques focus on minimizing adhesion, thus offering an effective alternative for managing hydrate-related problems. The use of hydrophobic and anti-hydrate surfaces with robust mechanical properties ensures the durability of oil and gas pipelines in extreme environments. However, significant challenges remain, including an insufficient understanding of the mechanisms underlying adhesion processes, the lack of standardized performance measurement methods, and inadequate evaluation of these techniques in realistic industrial contexts. These aspects underscore the need for further research to optimize the use of these approaches in practical applications.

Conflicts of Interest

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

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