Research Article

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# Synergistic Performance of Pectin with Monoethylene glycol as Environment Friendly and Sustainable Hydrate Inhibitor

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## ABSTRACT

This study evaluates pectin as a biodegradable, kinetic hydrate inhibitor (KHI) and examines its synergistic action with monoethylene glycol (MEG) for hydrate management. Hydrate inhibition was assessed from the induction time (IT) for methane hydrate formation at pectin concentrations of 0.25 wt%, 0.5 wt%, and 1 wt%, using methane at 7.6 MPa and a hydrate equilibrium temperature of 10.45 °C. The system was cooled at a constant rate of 1°C per hour below the hydrate equilibrium temperature. Pectin showed slightly lower inhibition performance than KHIs, but its biodegradable and sustainable nature indicates potential for reducing dependence on conventional non-biodegradable inhibitor polymers. Blending pectin with MEG improved the inhibition performance, demonstrating a synergistic effect that became pronounced with increasing MEG concentration. The blend provides an environmentally conscious approach for controlling hydrate formation in oil and gas flowlines, where chemical discharge and persistence are important considerations. The combination of a renewable biopolymer and MEG offers a promising basis for developing more sustainable hydrate-management strategies, while supporting further investigation of biodegradable materials for environmentally safer deep-sea flow assurance applications.

**Keywords:** Oil and gas; Hydrate management; Biodegradable materials; Pectin; Monoethylene glycol; Environmental sustainability.

## 1. INTRODUCTION

Reliable oil and gas transmission is essential for efficient and safe operation of production systems. Offshore flowlines frequently operate under temperatures and pressures that favor gas-hydrate formation; consequently, hydrate accumulation can restrict flow and cause blockage, pressure buildup, and potential pipeline damage (Mahmoud *et al.* 2014). Such incidents can interrupt production and increase maintenance requirements, making effective hydrate inhibition important for flow assurance. From an environmental perspective, the selection of inhibitors is also important because chemicals introduced into offshore production systems may persist, accumulate, or require additional handling after use. Therefore, developing effective inhibitors with improved environmental compatibility is an important aspect of sustainable flow-assurance practice. Fig. 1 depicts a hydrate plug formed during one of our experiments. This perspective is consistent with the broader objective of environmental nanotechnology research, where material selection, functional performance, resource efficiency, and environmental compatibility are considered together when developing technologies for industrial and energy-related applications. under demanding offshore operating conditions.

The industry commonly injects methanol or glycol to suppress hydrate formation (Anderson and Prausnitz 1986); these compounds are generally classified as thermodynamic hydrate inhibitors (THIs). When added to the production stream, THIs shift hydrate equilibrium toward higher pressures and lower temperatures. However, effective inhibition can require substantial THI concentrations, commonly 10–40 wt%, to achieve a meaningful reduction in equilibrium temperature (Wu *et al.* 2007). The purchase, transportation, storage, and recovery of large quantities of THIs increase operational cost and resource consumption (Chin and Srivastava, 2018). Their flammability and toxicity can also create handling and environmental concerns. Low-dosage kinetic hydrate inhibitors (KHIs) therefore offer an attractive route for reducing chemical consumption while maintaining flow assurance. From a materials perspective, the development of biodegradable inhibitor systems is particularly relevant because it can combine functional hydrate control with reduced environmental persistence.

Over the past two decades, KHIs, typically water-soluble polymers derived from crude-oil petrochemical fractions, have been developed, tested, and deployed by industry (Fu *et al.* 2001; Frostman *et al.* 2003; Glénat *et al.* 2004; Swanson *et al.* 2005; Wang *et al.* 2013). These materials generally delay hydrate formation and slow hydrate-crystal growth (Carver *et al.* 1995; O'Reilly *et al.* 2011). Proposed inhibition mechanisms include hydrogen bonding between KHI functional groups and water molecules, which can interfere with the formation of stable hydrate nuclei. However, these mechanisms remain incompletely understood because of the complexity of molecular interactions. This highlights the importance of investigating alternative functional materials whose molecular structure can provide hydrate inhibition while offering improved environmental compatibility and biodegradability.



**Fig. 1. Methane hydrate plug**

MacDonald *et al.* (2006) reported three field applications of KHIs for hydrate inhibition. The first application involved an offshore UK flowline connecting three gas platforms. The 69 km flowline experienced 4–8 °C subcooling, defined as the difference between hydrate-equilibrium temperature and minimum operating temperature. Previously, triethylene glycol (TEG) and MEG were used together as THIs, but frequent shutdowns occurred because of MEG/TEG regeneration problems, equipment failures, and requirements for maintenance and hydrate removal. In 1997, a KHI product was introduced, after which no new hydrate-formation problems were documented. The operator reported substantial first-year cost savings from KHI use. A second application involved a 17 km subsea pipeline connecting an autonomous platform to an onshore terminal, where low subcooling of 1–5 °C was encountered. KHI application prevented reported hydrate plugging until the end of field life. In the third application, hydrates occasionally formed in water-injection lines of an onshore system with water-reinjection wells, under subcooling of 7–13 °C. A KHI was recommended for the operating conditions, and no subsequent hydrate formation was observed in the injection lines. Although many KHIs are reported to have low toxicity (Gad *et al.* 1987; Fu *et al.* 2002; Exon 2006), their limited biodegradability remains an environmental concern (Magnusson et al. 2018). Incidents associated with the persistence of polymeric KHIs have been reported (Exon 2006; Eubeler *et al.* 2009; Eubeler *et al.* 2010; Lin *et al.* 2017; Farhadian *et al.* 2019), and restrictions have therefore been imposed in some locations because of non-biodegradability (Kelland, 2018). These concerns support continued development of biodegradable, environment-friendly, and sustainable hydrate-inhibitor materials (Singh and Suri, 2020; Singh and Suri, 2021; Singh and Suri, 2022; Singh *et al.* 2023b; Singh and Suri, 2023; Singh *et al.* 2023a).

Accordingly, the present study evaluates pectin as a biodegradable hydrate-inhibiting material and examines its synergistic performance with MEG. Pectin is a naturally occurring polysaccharide found within the cellular structures of plants and is commercially obtained mainly from apple and citrus fruit rinds and pulp (Colin, 1990). Its molecular structure is shown in Fig. 2. Pectin consists predominantly of galacturonic-acid units with methyl-ester groups distributed along the chain and generally has a molecular weight of 50,000–150,000 g/mol (Dorota *et al.* 2003). Its natural origin and biodegradability make it attractive for developing more environmentally compatible functional materials. Pectin has demonstrated more than 90% degradation within 28 days according to the OECD 306 test (Pereira *et al.* 2021) and has also been reported to be non-toxic. In addition, pectin exhibits binding activity toward toxic cations such as lead and mercury (Rudolf, 1982), indicating potential environmental relevance beyond hydrate control. The hydroxyl groups in its side chains can form hydrogen bonds with water molecules and thereby delay hydrate formation (Xu *et al.* 2016; Yaqub *et al.* 2019; Yaqub *et al.* 2021). The present work therefore focuses on the functional performance of this renewable biopolymer and its interaction with MEG as a route toward more sustainable hydrate-management chemistry for offshore applications.

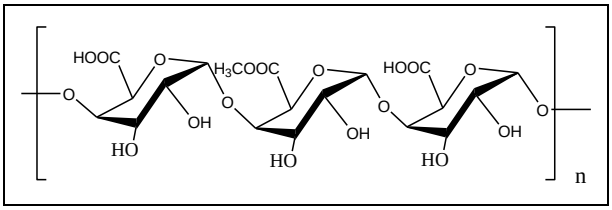



**Fig. 2: Molecular structure of pectin**

## 2. METHODOLOGY

Standard constant cooling rate tests were carried out in an autoclave to form methane hydrates and to examine the hydration-inhibiting effectiveness of individual and synergistic mixtures of pectin with MEG. The induction time was computed from the experimental data.

### 2.1 Material and Source

All chemicals tested were obtained from TCI chemicals.

### 2.2 Apparatus Used

An autoclave at high pressure with cooling capacity and SCADA system was used in all the tests, as shown in Fig. 3. The equipment details were covered in depth in our earlier work (Singh and Suri 2022b). In summary, the cell volume is 0.5 L that can be pressurized up to 200 bars and cooled down to -10 ℃ at different constant cooling rate such as, 1 ℃/hour. Gas-water mixture stirring at a variable rate was provided using a magnetic stirrer up to a stirring speed of 1000 rpm.

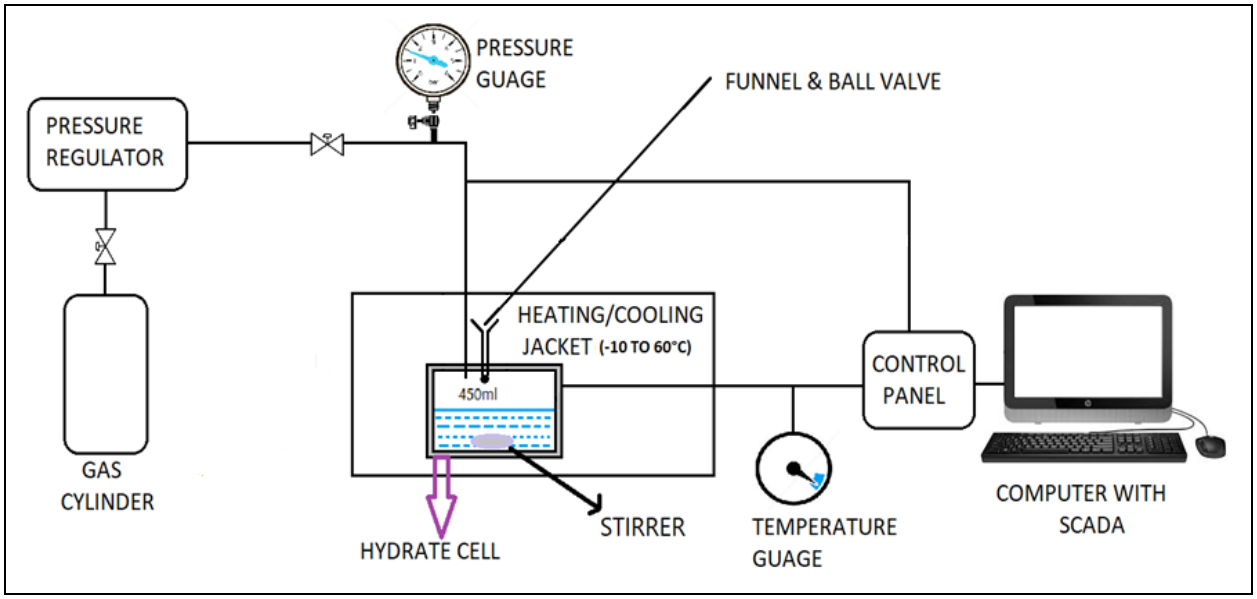



**Fig. 3: Diagram of the autoclave setup (Singh and Suri, 2022b) for hydrate formation and inhibitor performance evaluation**

### 2.3 Experimental Procedure

Hydrate formation and inhibitor testing was done using a constant cooling rate procedure (Singh and Suri, 2020; 2021). The detailed constant cooling experimental process can be accessed from our previous work (Singh and Suri, 2022). Figure 4 illustrates a standard plot depicting the recorded test parameters, including temperature, pressure, and time, during a consistent cooling experiment. In each experimental trial, we combine 150 mL of distilled water with varying concentrations of pectin and industrial hydrate inhibitors to create distinct blend ratios. For a distilled water-methane system at 7.6 MPa the hydrate equilibrium temperature ($T_{eq}$) is equal to 10.45 ℃ as found from CSM-Gem (Colorado school of Mines Gibbs Energy Minimization) research free software.

First, the cell was filled with distilled water and the temperature was reduced to the hydrate equilibrium temperature. Pure methane was introduced and pressure was increased to 7.6 MPa. The cell was then sealed, and a stirrer was used to stir the distilled water methane mixture at a speed of 600 rpm. Once the pressure and stirring were stabilized, cooling at a constant rate of 1℃/hour was applied. Pressure and temperature readings with time were recorded by SCADA until at least 0 ℃ is reached. Gradual temperature reduction causes pressure to reduce until hydrate formation. However, a sudden pressure reduction is observed at hydrate nucleation temperature due to the entrapment of free-pressured gas in the hydrate crystals. The time taken for hydrate formation from the equilibrium temperature is termed as induction time (IT) which is calculated from equation 1.

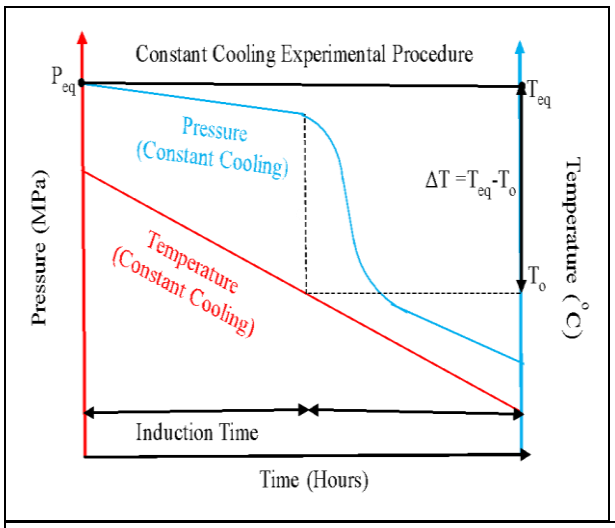



**Fig. 4: General pressure and temperature curves during constant cooling hydrate formation/inhibition experiments**

$$Change\ in\ induction\ time\ (\%) = \frac{IT_{(blend)} - IT_{(Pure\ KHI)}}{IT_{(Pure\ KHI)}} * 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

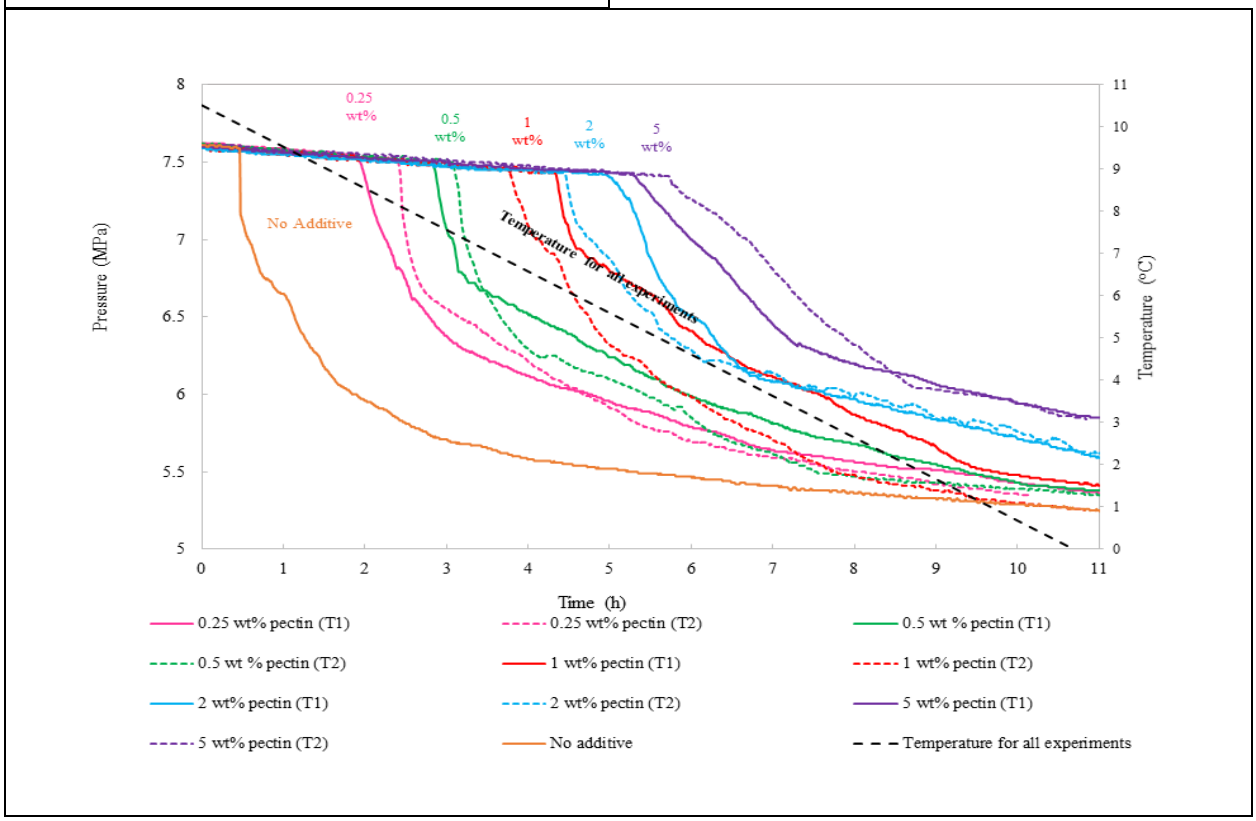



**Fig. 5: General pressure and temperature curves during constant cooling hydrate formation/inhibition**

Temperature data is averaged in the plots for clarity as it is almost the same for each experiment.

**Table 1. Induction time details for pectin at different concentrations**

| E. No. | KHI | Conc (wt%) | $T_{eq}$ (°C) | Test No. | $T_o$ (°C) | IT (h) | Avg IT (h) | IT std dev (h) |
|---|---|---|---|---|---|---|---|---|
| 1 | Pectin | 0 | 10.45 | T-1 | 8.54 | 1.91 | 2.13 | 0.30 |
| 2 | | 2 | | T-2 | 8.11 | 2.34 | | |
| 3 | | 5 | | | | | | |
| | | 0 | | T-1 | 7.63 | 2.82 | 2.92 | 0.15 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 4 | | 5 | | T-2 | 7.42 | 3.03 | | |
| 5 | | 1 | | T-1 | 6.11 | 4.34 | 4.04 | 0.42 |
| 6 | | | | T-2 | 6.71 | 3.74 | | |
| 7 | | 2 | | T-1 | 5.40 | 5.05 | 4.75 | 0.41 |
| 8 | | | | T-2 | 6 | 4.45 | | |
| 9 | | 5 | | T-1 | 4.71 | 5.74 | 5.47 | 0.38 |
| 10 | | | | T-2 | 5.25 | 5.2 | | |

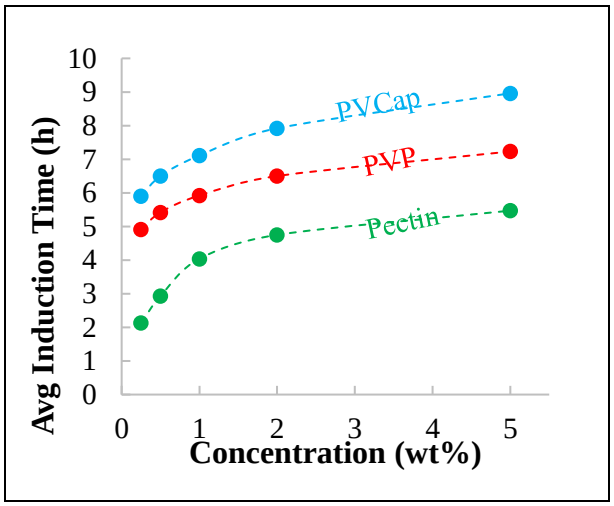



**Fig. 6: Comparison of the average Induction Times (IT) for pectin, polyvinyl pyrrolidone (PVP) and poly(N-vinylcaprolactam (PVCap) at different concentrations (Singh and Suri, 2022a; Singh *et al.* 2023c)**

## 3. RESULTS AND DISCUSSION

### 3.1 KHI Performance of Pectin

Experiments were performed at 0.25, 0.5, 1, 2 and 5 wt% of pectin for performance benchmarking. Experimental results measured for each of the individual inhibitors are presented in Fig. 5. It was observed that upon increasing the concentration of the different inhibitors, there was an increase in the IT.

From Fig. 6, it can be observed that performance of pectin is slightly slower than commercial KHI. It still demonstrates promising potential as a viable alternative to traditional non-biodegradable and unsustainable KHIs.

### 3.2 KHI performance of pectin - MEG blends

After obtaining the results for pectin as a standalone kinetic hydrate inhibitor, the groundwork was laid for investigating the potential synergy in hydrate inhibition between MEG and pectin. To explore this synergy, four distinct concentrations of MEG ranging from 0.5 % to 20 % by weight were combined with 0.25 % pectin for testing purposes. Figure 7 depicts the outcomes of two separate sets of hydrate formation experiments, showcasing the variations in pressure and temperature over time. The repeated tests are indicated by solid and dashed lines of the same color. Table 2 provides a summary of the results, including the hydrate nucleation temperature (To) and induction times (ITs) for both sets of experiments, along with their corresponding averages and standard deviations.

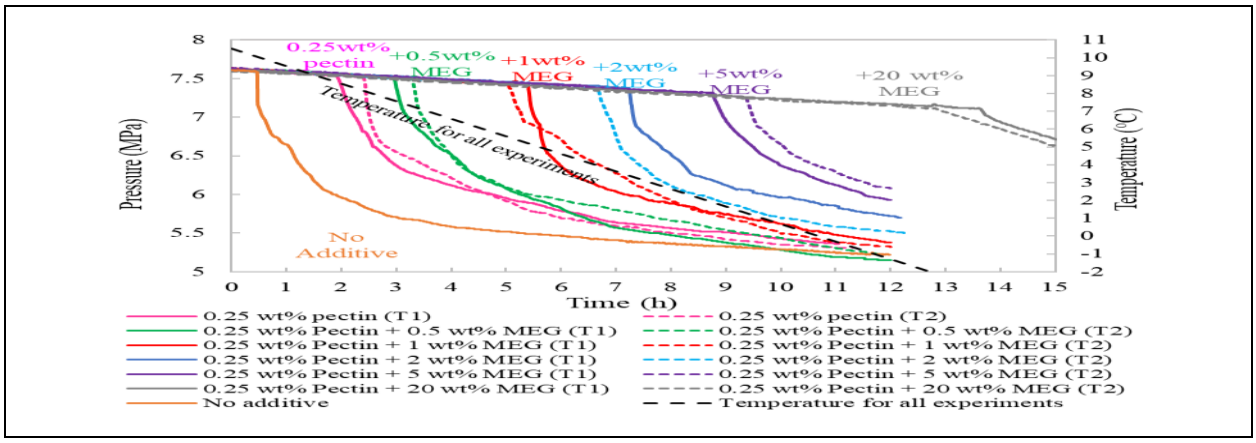



**Fig. 7: Hydrate formation results for pectin blended MEG**

**Table 2. Average To and IT values for pectin MEG blends**

| S. No. | KHI | Conc of MEG (wt %) | Blend $T_{eq}$ (°C) | Test No. | Blend $T_o$ (°C) | Average $T_o$ (°C) | Blend IT (h) | Average IT (h) | IT (h) std dev |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Pectin (0.25 wt%) | 0 | 10.45 | T-1 | 8.54 | 8.32 | 1.91 | 2.13 | 0.30 |
| 2 | | | | T-2 | 8.11 | | 2.34 | | |
| 3 | | 0.5 | 10.35 | T-1 | 7.52 | 7.33 | 2.83 | 3.02 | 0.26 |
| 4 | | | | T-2 | 7.15 | | 3.2 | | |
| 5 | | 1 | 10.24 | T-1 | 5.06 | 5.25 | 5.18 | 4.98 | 0.28 |
| 6 | | | | T-2 | 5.45 | | 4.79 | | |
| 7 | | 2 | 10.03 | T-1 | 3.20 | 3.50 | 6.83 | 6.53 | 0.42 |
| 8 | | | | T-2 | 3.80 | | 6.23 | | |
| 9 | | 5 | 9.38 | T-1 | 1.71 | 1.39 | 7.67 | 7.99 | 0.45 |
| 10 | | | | T-2 | 1.08 | | 8.3 | | |
| 11 | | 20 | 4.95 | T-1 | -3.19 | -2.83 | 8.14 | 7.78 | 0.49 |
| 12 | | | | T-2 | -2.47 | | 7.42 | | |

Table 2 shows the influence of varying MEG concentrations with a pectin solution of 0.25 wt% on the average temperature at which hydrate inhibition occurs (IT). When the mixture includes 0.5 wt% MEG along with 0.25 wt% pectin, it results in a reduction of the average temperature at which hydrate formation begins (To) to 7.33°C, subsequently resulting in an average IT of 3.02 hours. Likewise, increasing the MEG concentration to 1 wt% leads to a decrease in the average To to 5.25°C, thereby elevating the average IT to 4.98 hours. When 2 wt% MEG is combined with 0.25 wt% pectin, the average temperature for hydrate formation stabilizes at approximately 3.5°C, leading to an average IT of 6.53 hours. Furthermore, when 5 wt% MEG is mixed with 0.25 wt% pectin, the To diminishes to 1.39°C, resulting in an IT of 7.99 hours. Finally, introducing 20 wt% MEG to 0.25 wt% pectin exhibits a significant reduction in nucleation temperature (-2.83). However, the resulting IT remains almost unchanged when compared to the scenario with 5 wt% MEG. Hence, it can be concluded that increasing the concentration of MEG solvent beyond 5 wt% does not substantially enhance the IT of pectin.

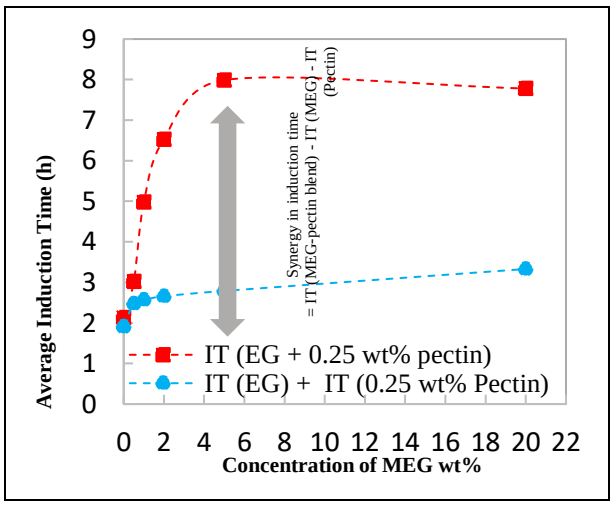



**Fig. 8. Synergistic hydrate inhibition by the pectin-MEG blends**

Figure 8 illustrates both the blend ITs and the cumulative ITs of separate MEG and pectin components. The presence of synergy between the chemicals for hydrate inhibition can be determined by comparing the blend IT with the sum of individual ITs. The figure reveals that synergy levels are relatively low when MEG constitutes less than 0.5 wt% of the blend, but they rise significantly as MEG concentration increases, reaching a peak at 5 wt%. Subsequently, there is a slight reduction in synergy, although it remains substantial.

## 4. CONCLUSIONS

The experimental results demonstrate that pectin can function as a biodegradable, plant-derived kinetic hydrate inhibitor, although its inhibition performance was slightly lower than that of commercial KHIs. Its renewable origin, biodegradability, and potential environmental compatibility make it a promising alternative to persistent synthetic inhibitor polymers. When combined with MEG, pectin showed a synergistic improvement in hydrate inhibition, with performance increasing as MEG concentration increased up to 5 wt%. Beyond 5 wt% MEG, further addition produced little improvement in induction time, although hydrate nucleation temperature decreased. The pectin–MEG system therefore combines effective hydrate control with a more sustainable materials perspective for offshore flow-assurance applications. These findings support investigation of biodegradable biopolymers and their synergistic combinations as responsible hydrate-management materials, including their long-term degradation, marine environmental behavior, and performance under field conditions.

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## CONFLICT OF INTEREST

Authors have no conflict of interest.

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