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# Rheological properties of $CO_2$ hydrate slurry produced in a stirred tank reactor and a secondary refrigeration loop

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ABSTRACT

The aim of this paper is to present the rheological properties of $CO_2$ hydrate slurry for a use as secondary fluids in refrigeration systems. A set-up composed of a stirred tank reactor and a circulation loop was used to study $CO_2$ hydrate slurry formation and flowing. Rheological properties of $CO_2$ hydrate slurries circulating in the loop were determined by the capillary viscometer method. The results show a shear thinning behaviour of the $CO_2$ hydrate slurries for a solid fraction up to 22%. This behaviour is correlated by an Ostwald-de-Waele empirical equation, which takes into account the hydrate fraction of the slurry. The apparent viscosity of $CO_2$ hydrate slurry was estimated from the model and a good agreement was found with the experimental data. A comparison with literature shows the importance of using a stirred reactor for slurry homogenisation, which allows the decrease of the apparent viscosity of the slurry.

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# Propriétés rhéologiques d’un coulis d’hydrate de $CO_2$ produit dans un bac sous agitation et une boucle secondaire

*Mots clés :* hydrates de dioxyde de carbone ; changement de phase ; coulis ; rhéologie ; écoulement multiphasique ; froid

## 1. Introduction

The use of a secondary circuit for cold distribution is an alternative to reduce the quantity of traditional refrigerants. The secondary refrigerant distributes cold using alternative environment-friendly fluids and the primary circuit using traditional greenhouse-effect refrigerants is then confined and minimized. However, secondary refrigeration requires

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**Nomenclature**

*Notation*

| | |
|---|---|
| $D$ | Pipe diameter (m) |
| $k$ | Consistency index (Pa s) |
| $L$ | Tube length (m) |
| $n$ | Behaviour index |
| $P$ | Pressure (MPa) |
| $Q$ | Volume flow rate ($m^3\ s^{-1}$) |
| $R$ | Pipe radius (m) |
| $T$ | Temperature (K) |
| $U$ | Fluid velocity ($m\ s^{-1}$) |

*Greek letters*

| | |
|---|---|
| $\Delta P$ | Pressure drop (Pa) |
| $\dot{\gamma}_w$ | Shear rate at the wall ($s^{-1}$) |
| $\phi_s$ | Volume fraction of hydrate (vol.%) |
| $\tau_0$ | Minimal shear or yield stress (Pa) |
| $\tau_w$ | Shear stress at the wall (Pa) |
| $\mu_{app}$ | Apparent viscosity (Pa s) |

the use of heat exchangers and additional circulating pumps which generate exergy losses in the system. In order to limit these losses, and thus to improve the system efficiency, it is possible to transport the cold via a two-phase (solid–liquid) secondary refrigerant (TPSR), composed of solid particles in suspension able to store a large amount of energy by latent heat. Moreover, the temperatures of TPSR are stable, which minimizes the thermodynamic irreversibility in the exchangers. Ice slurries (suspension of ice crystals in a carrying liquid phase) are the most current TPSRs (Guilpart et al., 2006), but their industrial development is limited by the generators based on mechanical processes (scraped or brushed surface exchangers) which are often power-limited. Clathrate hydrate slurries (CHS) may also be used as TPSRs. Gas hydrates are crystalline solids resulting from the arrangement of water molecules linked by hydrogen bonds constituting cages around stabilizing gas molecules. They were primarily studied in oil and gas industry since they could appear in the pipelines due to natural gas (methane, ethane) and water coexistence under appropriate pressure and temperature conditions and then provoke pipeline plugging. Studies on hydrate formation, remediation, and prevention are still numerous (Sloan, 2005). CHS are also investigated for natural gas capture processes, where gas is injected in liquid water-oil emulsion to form hydrates (Fouconnier et al., 2002; Huang et al., 2009), and for $CO_2$ capture and sequestration (Li et al., 2009a, 2009b; 2011).

The first studies on rheology for hydrate slurries were performed by Pinder (1964) on a suspension of hydrogen sulfide hydrate and tetrahydrofuran ($H_2S$-2THF-17$H_2O$). Using a rotational viscometer, the author showed the thixotropic behaviour of the slurry with a gel structure at very low hydrate concentration. Later, hydrate-slurry flow properties were

**Table 1 – Work on rheology of hydrate slurries. *work for refrigeration applications; in bold: our work; HC: hydrocarbon; TA: surfactant; AA: antiagglomerant; $\phi_s$: hydrate volume fraction = $V_{solid}/(V_{solid} + V_{liquid})$; R141b: refrigerant HCFC 141-b; OdW: Ostwald-de-Waele; S-thin.: Shear thinning; S-thicken.: Shear thickening; HB: Herschel-Bulkley.**

| Authors | Hydrate | Liquid | Viscometer | $\phi_s$ | Behaviour |
|---|---|---|---|---|---|
| Pinder (1964) | $H_2S$ + THF | Aqueous | Rotating | <0.01 | Thixotropic gel, in 76 h $\mu_{app}$: decrease to 60 at 23 mPa s |
| Austvik and Bjorn (1992) | HC | Organic | Rotating | – | No measure of $\tau_w$ |
| Fukushima et al. (1999)* | TBAB | Aqueous | Capillary | 0.22–0.31 | OdW S-thin.: $\mu_{app}$: 30–2000 mPa s |
| Andersson and Gudmundsson (1999) | HC | Organic + AA | Capillary | 0–0.1 | Bingham: $k$: 3.4–5.5 mPa s |
| Andersson and Gudmundsson, 2000 | $CH_4$ | Aqueous | Capillary | 0.01–0.1 | Bingham: $k$: 1–3.5 mPa s |
| Oyama et al. (2002) | $CO_2$ | Aqueous | Magnetic (Stress) | – | $\mu_{app}$ increase before nucleation, decrease after |
| Fidel-Dufour and Herri (2002) | HC | Organic + TA | Capillary | – | $\mu_{app}$ increase before nucleation, decrease after |
| Peysson et al. (2003) | HC | Organic | Capillary | 0.1–0.3 | OdW S-thicken. $n \approx 2$, $k \approx 2.10^{-3}$ mPa s |
| Darbouret et al. (2005)* | TBAB | Aqueous | Capillary | 0.04–0.53 | Bingham: $k$: 8–170 mPa s |
| Xiao et al. (2006)* | TBAB | Aqueous | Capillary | 0–0.16 | OdW S-thin.: $\mu_{app}$: 4–42 mPa s |
| Fidel-Dufour et al. (2006) | $CH_4$ | Organic + AA | Capillary | 0.07–0.18 | Newtonian: 2.5–3.5 mPa s |
| Delahaye et al. (2008)* | **$CO_2$** | **Aqueous** | **Capillary** | **0.04–0.1** | **~ OdW S-thicken.** |
| | | | | **0.1–0.2** | **HB S-thin.: $\mu_{app}$: 4–42 mPa s (at 400 $s^{-1}$)** |
| Wang et al. (2008)* | R141b | Aqueous | Capillary | 0.1–0.68 | OdW S-thicken.: $\mu_{app}$: 1.1–1.7 mPa s (at 400 $s^{-1}$) |
| Delahaye et al. (2011)* | **$CO_2$** | **Aqueous + TA** | **Capillary** | **0.04–0.1** | **Newtonian: $\mu_{app}$: 3.3–16.6 mPa s** |
| Ma et al. (2010)* | TBAB | Aqueous | Capillary | 0.06–0.2 | OdW S-thin.: $\mu_{app}$: 3–100 mPa s |
| Kumano et al. (2011)* | TBAB | Aqueous | Ubbelohde | 0.02–0.25 | OdW S-thin.: $\mu_{app}$: 2–5 mPa s |
| Hashimoto et al. (2011)* | TBAB | Aqueous | Plate | 0.12–0.7 | OdW S-thin.: $\mu_{app}$: 3.5–1000 mPa s |
| | TBAF | Aqueous | Plate | 0–0.42 | OdW S-thin.: $\mu_{app}$: 10–750 mPa s |
| Clain et al. (2012)* | **TBPB** | Aqueous | **Capillary** | **0–0.28** | **OdW S-thin.: $\mu_{app}$: 4-41 mPa s** |
| Webb et al. (2012) | HC | Organic | Rotating | 0.2–0.45 | HB S-thin.: $\mu_{app}$: 500–3000 mPa s |

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