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Review

A comprehensive review on CO₂ thickeners for CO₂ mobility control in enhanced oil recovery: Recent advances and future outlookEmanuel X. Ricky^{a,b}, Grant Charles Mwakipunda^a, Edwin E. Nyakilla^a, Naswibu A. Kasimu^{a,b}, Chao Wang^c, Xingguang Xu^{a,*}^a Key Laboratory of Tectonics and Petroleum Resources, Ministry of Education, China University of Geosciences (Wuhan), Wuhan 430074, China^b Department of Chemistry, College of Natural and Applied Sciences, University of Dar es Salaam, P.O. Box 35061, Dar es Salaam, Tanzania^c Engineering Technology Institute, Research Institute of Petroleum Exploration & Development, Beijing 100083, China

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ABSTRACT

Carbon dioxide (CO₂) has been utilized to recover the residual oil from the geological reservoirs through enhanced oil recovery (EOR) methods for over 50 years. Despite its long history of success as an EOR technique, CO₂ flooding recovers only about 20–40% of the original oil in place (OOIP) from the geological reservoirs. The small amount of oil recovered by CO₂ flooding is associated with the low viscosity of CO₂ injected into the reservoir, resulting in CO₂ viscous fingering, CO₂ gravity override and unfavourable mobility. To address these problems, the CO₂ viscosity needs to be enhanced considerably using CO₂ thickeners or viscosifiers. Despite more than five decades of intensive research work in formulating and identifying effective CO₂ thickeners such as polymers, surfactants, small molecules and nanoparticles; as yet none of these chemicals can be regarded as effective CO₂ thickeners for EOR field applications. Thus, CO₂ thickener is an interesting research topic for future studies to come up with effective and affordable CO₂ thickeners for EOR field applications. This article presents the recent developments in CO₂ thickening technologies in EOR. Furthermore, the CO₂ thickening mechanisms, screening criteria, field scale applications, challenges and future research directions on CO₂ thickeners are evaluated.

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Introduction

Oil and natural gas are very important sources of energy in the world to produce heat, transportation fuels and electricity [1]. Currently, global energy consumption is increasing rapidly while the existing oil and natural gas reserves are being exhausted day to day [2–4]. This has necessitated the oil and gas industry to develop new methods to recover more oil and gas from matured oil fields or conventionally inaccessible reservoirs [2,5,6]. The oil recovery processes have been typically divided into three stages such as primary, secondary and tertiary oil recovery [7–9]. Typically, only a fraction (5–20%) of the original oil in place (OOIP) can be recovered from the geological reservoirs using primary oil recovery methods [5,10], whereas the secondary oil recovery methods can only exploit about 20–40% of the OOIP [11,12]. When the secondary oil recovery methods are no longer effective, the artificial methods of oil recovery called tertiary oil recovery or enhanced oil recovery (EOR) are utilized to increase the recovery of the residual oil from the geological reservoirs beyond the limit recoverable by primary and secondary oil recovery methods [7,13].

EOR involves the use of external materials or energy to recover the oil that cannot be generated economically using traditional methods or the natural energy of the reservoirs [8,14,15]. The EOR techniques help to boost the reservoir energy, reduce the interfacial tension (IFT) between the displacing fluid (CO₂) and displaced fluid (oil), increase the viscosity of the displacing fluids, reduce the capillary forces and reduce the oil viscosity [16–19]. However, the primary objective of EOR techniques is to improve the volumetric displacement efficiency of the residual oil in the reservoir via the injection of materials that are not usually present in the reservoir and increase the oil production at an economically feasible production rate [7,20,21]. The EOR techniques are mainly divided into four major groups including chemical flooding, thermal flooding, gas flooding, and microbial-enhanced oil recovery (MEOR) [11,20,22,23]. Among these techniques, gas flooding and thermal flooding techniques are widely used because they are the most technically and economically viable EOR methods [14,24]. In gas flooding (i.e., CH₄, N₂ and CO₂), CO₂ flooding is the most popular and extensively utilized EOR technique [2,25–33]. Despite its historical success, the CO₂ flooding method yet does not recover completely the original oil in place [34]. The insufficient amount of oil recovered by the CO₂ flooding method is associated with the low viscosity and density of CO₂ injected into the reservoir [35,36]. The low density and viscosity of CO₂ results in CO₂ viscous fingering, CO₂ gravity override and unfavourable mobility which reduces the oil recovery [37–39]. These problems

have prompted researchers in the oil industry to develop mitigation methods to enhance the performance of CO₂ flooding in EOR. Currently, the application of CO₂ thickeners or viscosifiers such as polymers, surfactants, small molecules and nanoparticles is the only known mitigation method to solve the problem of low density and viscosity of CO₂ [40]. The CO₂ thickeners are not only aimed at increasing the viscosity of CO₂ but also to improve the mobility of CO₂ during EOR and increase the oil recovery.

The objective of this work is to review previous and current research developments in CO₂ thickening technologies and provides future research directions on CO₂ thickeners. The uniqueness of this work is that the mechanisms of CO₂ thickening, screening criteria for selecting or designing CO₂ thickeners, challenges facing CO₂ thickening technologies and future research directions on CO₂ thickeners are described in detail. Besides, the CO₂ thickeners are classified based on their chemical composition or functional groups, and both available laboratory and field research findings on CO₂ thickeners are reported and analysed. Therefore, this review paper is expected to provide a comprehensive understanding of CO₂ thickeners or CO₂ thickening technologies and serves as a guide for selecting or designing effective and affordable CO₂ thickeners for EOR field applications. The main sections of this article are presented using a flowchart as shown in Fig. 1.

Background of CO₂-EOR

CO₂ has been employed to recover the residual oil from geological reservoirs for more than five decades [10,41–43]. The first patent on CO₂-EOR technology was awarded to Whorton, Brownscombe and Dyes of the Atlantic Refining Company in 1952 [44]. The first commercial CO₂-EOR field application was pioneered in the early 1970s in the Kelly-Snyder oil field in West Texas and continues to be the world's largest CO₂ miscible flooding project until today [45–49]. The amount of CO₂ currently used for EOR operations ranges from 65 to 72 million tonnes per year [46]. The number of CO₂-EOR projects in the United States of America (USA) alone between 1972 and 2014 was reported to be 128 projects, of which 104 were successful by 81% [45]. The USA produces a significant amount of its oil using the CO₂-EOR method. Currently, 280,000 barrels of oil per day are produced from CO₂-EOR, which is over 5% of the total US crude oil production [50]. The next generation CO₂-EOR target is to increase oil production to 67–137 billion barrels [46]. To meet this target, a substantial improvement in the CO₂ flooding method is inevitable to recover more oil beyond 10–20% of the OOIP currently being recovered by the conventional CO₂-EOR technology [49,50]. Recently, the CO₂ flooding method

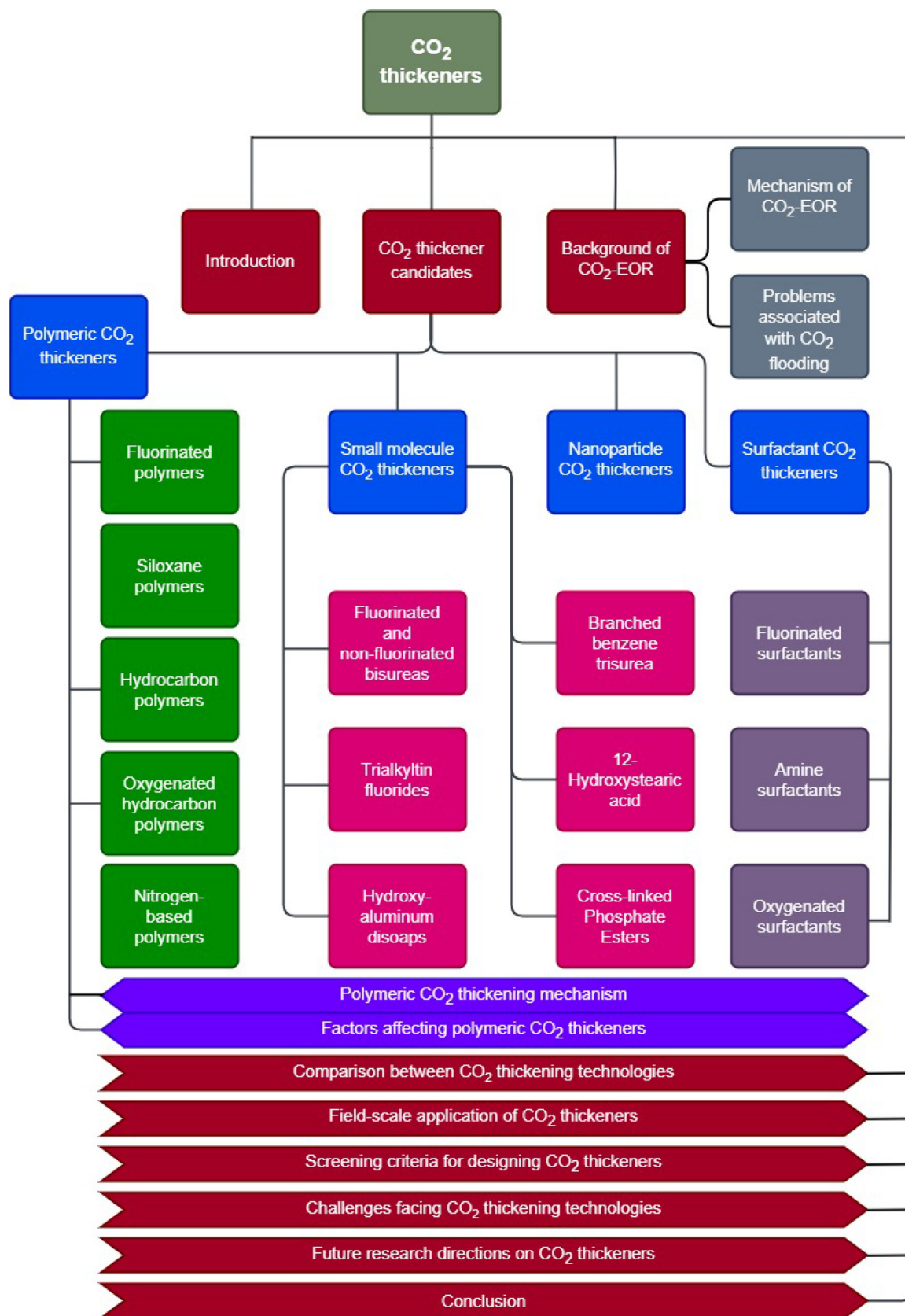


Fig 1. Flowchart showing the main sections discussed in this work.

has become technically and economically very attractive worldwide [5,14,50,51]. The important properties that make CO₂ more attractive and an ideal gas for EOR operations include its availabil-

ity in large quantities from natural and anthropogenic sources, it is less expensive, highly soluble in crude oil, and non-flammable and non-toxic gas [52–61]. Also, the CO₂ exists in a gaseous state at

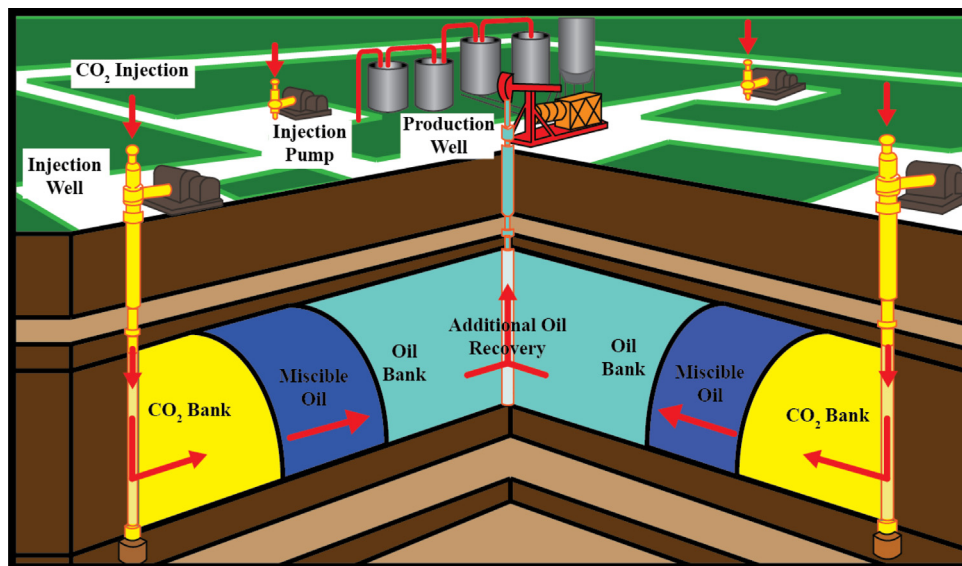


Fig. 2. Mechanism of CO₂ miscible flooding during enhanced oil recovery.

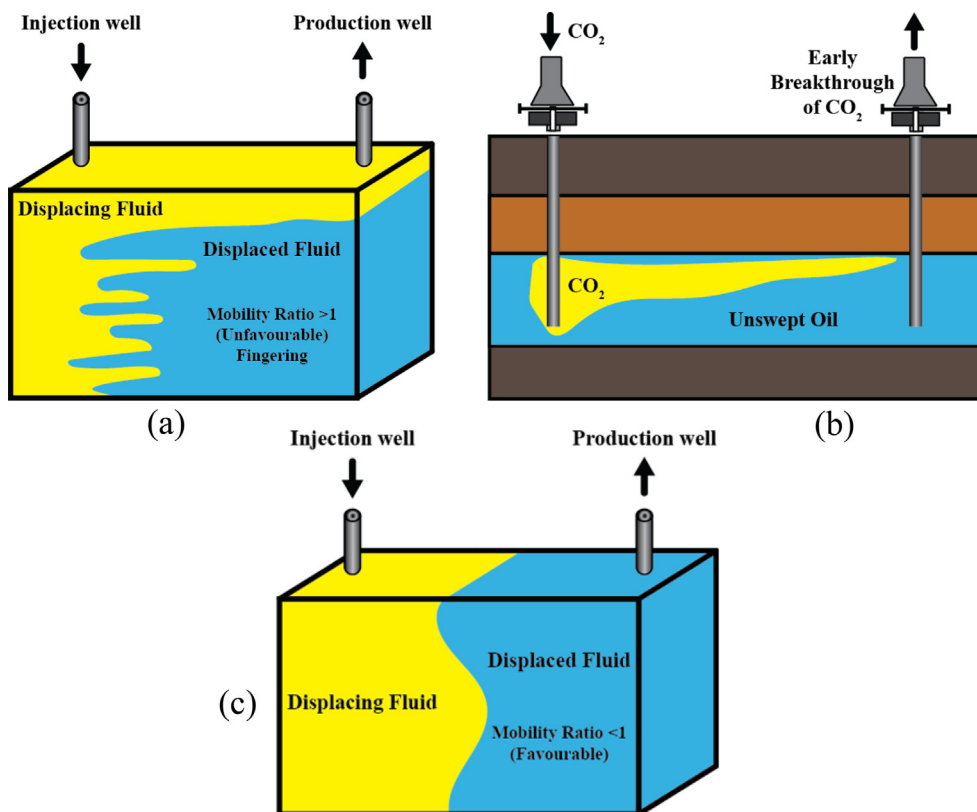


Fig. 3. A scheme of CO₂ viscous fingering (a), early CO₂ breakthrough (b) and controlled CO₂ mobility (c).

standard temperature and pressure; thus, it can be easily separated from the crude oil after the EOR operation by releasing the pressure.

Mechanism of CO₂-EOR

The performance of CO₂-EOR depends on crude oil composition, reservoir temperature and pressure. CO₂ flooding is commonly used for mobilizing light oil components from geological reservoirs

[62]. When CO₂ is injected into an oil reservoir, it tends to develop miscibility and becomes soluble with the residual oil in place [63]. The dissolution of CO₂ in crude oil leads to a reduction in crude oil viscosity and interfacial tension [5,64–66]. The dissolved CO₂ swells the oil which in turn increases the volume and relative permeability of the residual oil in the reservoir rock pores [65,67,68]. When the reservoir pressure is above the minimum miscibility pressure (MMP), the viscosity of the crude oil in the reservoir rock is significantly reduced by CO₂ saturation and the residual oil is

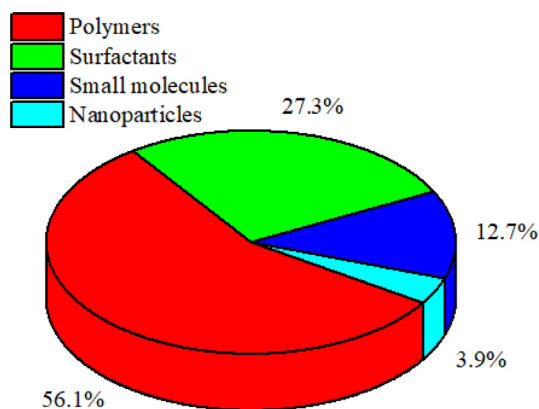


Fig. 4. Distribution of the research effort spent on various types of CO₂ thickeners over the past 50 years.

ultimately accelerated towards the production well by CO₂ [69] (Fig. 2).

Problems associated with CO₂ flooding

The most serious challenge that limits the wide applicability of CO₂ in EOR operations is the low viscosity of CO₂ [70,71]. The viscosity of liquid CO₂ at reservoir conditions ranges from 0.03 to 0.10 cp, while that of crude oil ranges from 0.1 to 50 cp [72–74]. Thus, this indicates that the viscosity of CO₂ is lower compared to that of crude oil by over one order of magnitude [75,76]. Since the relative permeability of CO₂ and crude oil are comparable in magnitude, the viscosity ratio between displacing fluid (CO₂) and displaced fluid (crude oil) leads to an unfavourable mobility ratio (mobility > 1) [52,77]. The unfavourable mobility ratio between CO₂ and crude oil results in CO₂ viscous fingering (Fig. 3a) and early CO₂ breakthrough (Fig. 3b) which reduces the oil recovery [40,78–81]. The mobility ratio between CO₂ and crude oil can be reduced or improved by enhancing the viscosity of CO₂ using CO₂ thickeners to the extent that the mobility < 1 (favourable) is achieved and the CO₂ viscous fingering is reduced (Fig. 3c) while the oil recovery is increased [27,82].

CO₂ thickener candidates

Several reservoir treatment technologies including the use of CO₂ thickeners have been suggested in previous studies for enhancing CO₂ flooding efficiency. The initiatives of finding appropriate CO₂ thickeners have started since the year 1970s until today [50,83]. Several attempts have been conducted in identifying suitable CO₂ thickeners which can improve the mobility ratio between CO₂ and crude oil during EOR operations. Heller and Taber [84] were the first researchers to study and report the findings on the use of direct thickeners for mobility control of liquid CO₂. In one of their studies, they tested 53 chemical compounds as CO₂ thickeners. Their findings revealed that only 17 chemical compounds out of 53 were soluble in CO₂ with minor viscosification [35,72,85]. In another attempt, Llave and coworkers have used entrainers/co-solvents such as n-decanol, ethoxylated alcohols, isooctane, 2-ethyl hexanol, hexane, acetone, ethanol, acetonitrile, ethyl acetate, ether and chloroform to increase the viscosity of CO₂ [75,86]. From their findings, they discovered that the entrainers alone are not effective CO₂ thickeners; however, if they are used together with other thickeners such as polymers they can increase the viscosity of CO₂ [87]. Generally, the basic approach to increase the viscosity of CO₂ is to use CO₂ thickeners which would dissolve in CO₂ at actual reservoir conditions [88–90]. Based

on the molecular structure of thickeners and their thickening mechanism, CO₂ thickeners are categorized as polymers, surfactants, small molecule compounds and nanoparticles [50,70,85,91]. The research effort spent on various types of CO₂ thickeners since 1970s is presented in Fig. 4 and implies that polymers are the most commonly studied and successful CO₂ thickeners as compared to other thickeners. This is because polymers form thermodynamically stable single-phase solutions with a viscosity slightly higher than that of crude oil at reservoir conditions [37,92,93]. Since the molecular structure and mechanism of action of one type of CO₂ thickener is different from other types, in this study each type of CO₂ thickener is treated separately as described in subsections 3.1 to 3.4.

Polymeric CO₂ thickeners

The application of polymeric thickeners is one of the fundamental methods to increase the viscosity of CO₂ [94]. The CO₂ thickening properties of polymeric compounds have been investigated continuously since the year 1970 s [50,83]. Several attempts have been made in finding appropriate polymeric CO₂ thickeners [66,70,75]. Enick and co-workers have published a comprehensive review based on the studies which have attempted to thicken CO₂ using several chemical compounds including polymers for more than four decades [50]. Previous literature reported that polymeric thickeners are more promising than other methods because of the stability of CO₂-polymer solution under the actual reservoir conditions [95,96]. Several polymeric compounds have been investigated as direct CO₂ thickeners [97]. However, polymers with heteroatoms such as oxygen (O), nitrogen (N), silicon (Si) and halogens (i.e., fluorine (F)) are reported as effective CO₂ thickeners because they are more CO₂-philic as compared to polymers with carbon and hydrogen atoms only [98,99]. Based on the presence of heteroatom in the polymer chain, in this study, the CO₂ polymeric thickeners are classified as fluorinated polymers, silicone polymers, oxygenated hydrocarbon polymers and nitrogen polymers as described in subsection 3.1.1 to 3.1.5. The molecular structures of the frequently investigated polymeric CO₂ thickeners are given in Table 1 and their experimental findings are discussed in subsections 3.1.1 to 3.1.5 and summarized in Table 2.

Fluorinated polymers

The polymers in this group are described by carbon-fluorine linkages and are capable of increasing the viscosity of CO₂ without a co-solvent [91,110]. DeSimone and co-workers were the first research group to demonstrate the ability of fluoro-polymers to thicken CO₂ without a need for co-solvents [100]. They found that the addition of 3.4 and 6.7 wt/vol% poly(1,1-dihydroperfluorooctyl acrylate) (PFOA, Mw = 1,400,000 g/mol, compound 1, Table 1) in neat CO₂ increased the CO₂ rich solution viscosity by 2.5 and 6 fold (Fig. 5), respectively at 31 MPa and 323 K. Currently, polyfluoroacrylates remain the most CO₂-soluble polymers and most effective CO₂ thickeners [110–112]. Nonetheless, the environmental concerns, high cost and high concentration of PFOA needed to thicken CO₂ make PFOA not practical for CO₂-EOR field applications [105].

In another attempt, Enick and colleagues [52,102] synthesized polyfluoroacrylate styrene copolymer (polyFAST, compound 2, Table 1) to improve the CO₂ thickening ability of polyfluoroacrylate polymers. The polyFAST was found to be soluble in CO₂, however, its solubility was observed to diminish as the styrene concentration increased. Their experimental studies revealed that the addition of 0.5 and 1 wt% polyFAST in CO₂ at 323 K and 34 MPa increased the CO₂ viscosity by 1.5 and 2.3 fold, respectively [102]. In comparison to PFOA, polyFAST was the most effective CO₂ thickener at low concentrations in the absence of a co-

Table 1
The molecular structures of polymeric CO₂ thickeners evaluated in this work.

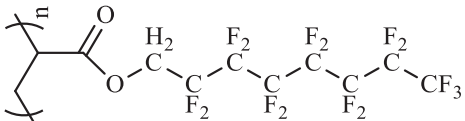
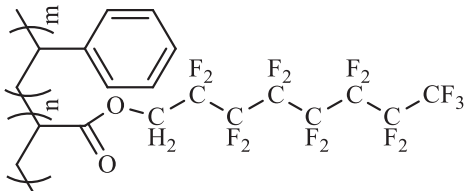
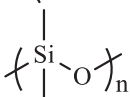
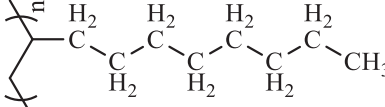
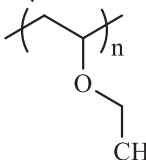
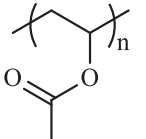
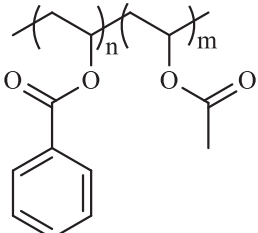
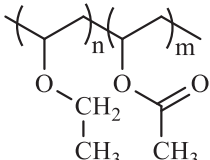
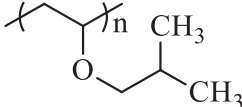
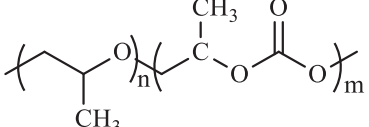
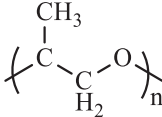

Compound	Molecular structure	Polymer name	Reference
1		Poly(1,1-dihydro-perfluorooctyl acrylate) (PFOA)	[100,101]
2		Poly(fluoroacrylate styrene) (PolyFAST)	[102,103]
3		Poly(dimethylsiloxane) (PDMS)	[104,105]
4		Poly(1-decene) (P-1-D)	[104]
5		Poly(vinyl ethyl ether) (PVVE)	[104,106]
6		Poly(vinyl acetate) (PVAc)	[105]
7		Poly(benzoyl-vinyl acetate) (PolyBOVA)	[105]
8		Poly(vinyl acetate-vinyl ethyl ether) (PVAEE)	[55]
9		Poly(iso-butyl vinyl ether) (Piso-BVE)	[95]
10		Poly(ether carbonate) (PEC)	[107,108]
11		Poly(propylene oxide) (PPO)	[106,109]
12		Poly(methyl acrylate) (PMA)	[106,108]

Table 1 (continued)

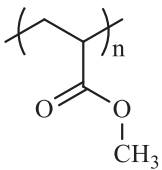
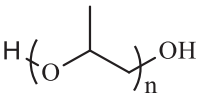
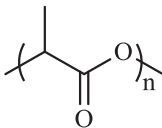
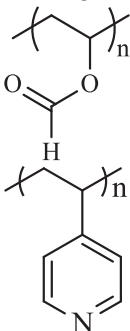
Compound	Molecular structure	Polymer name	Reference
13		Poly(propylene glycol) (PPG)	[108]
14		Poly(lactic acid) (PLA)	[106,108]
15		Poly(vinyl formate) (PVF)	[106,109]
16		Poly(4-vinyl pyridine) (P4VP)	[106]

Table 2

Summary of studies on the polymeric CO₂ thickeners discussed in this work.

Polymer shortened form	Molecular weight (g/mol)	Polymer concentration (wt%)	Co-solvent (wt%)	Experimental condition	Solubility in CO ₂	CO ₂ thickening ability	Reference
PFOA	1,400,000	3.4	No co-solvent	323 K & 31 MPa	Soluble	2.5 fold	[101,130]
PolyFAST	540,000	1.5	No co-solvent	298 K & 15 MPa	Soluble	19 fold	[130,140]
PDMS	197,000	4	20% Toluene	327 K & 17.2 MPa	Soluble	30 fold	[95]
PVEE	3,800	1.2–2	No co-solvent	377 K & 55 MPa	Soluble	1.2–2.1 fold	[95,105]
P-1-D	910	0.81–5	No co-solvent	377 K & 55 MPa	Soluble	1.2–2.77 fold	[95,105]
PVAc	500,000	2	23% Toluene	298 K & 64 MPa	Soluble	Increased by 70%	[103]
PolyBOVA	1,400,000	3.7	No co-solvent	298 K & 64 MPa	Soluble	1.8 fold	[130]
PVAEE	4,300	1.19–2.35	-	298 K	-	2–4 fold	[55,66]
Piso-BVE	4000	1.5	No co-solvent	377 K & 55 MPa	Soluble	1.23 fold	[95]
PEC	16,000	1	No co-solvent	295 K & 14 MPa	Soluble	Not significant	[130,132]
PPO	409	1	No co-solvent	306 K & 17.9 MPa	Highly soluble	1.25 fold	[72,133]
PMA	1,390	5	No co-solvent	298 K & 34.6 MPa	Soluble	Not significant	[98,109]
PPG	1000	1	No co-solvent	298 K & 31 MPa	Soluble	Not significant	[135]
PLA	128,500	5	No co-solvent	308 K & 140 MPa	Less soluble	No viscosity increase	[72,109]
PVF	125,000	5	No co-solvent	484 K & 240 MPa	Insoluble	No viscosity increase	[106,109]
PUD	32,500	4	No co-solvent	298 K & 34.5 MPa	Less soluble	2.7-fold	[73]
P4VP	160,000	0.7	No co-solvent	343 K & 55 MPa	Insoluble	No viscosity increase	[106]

solvent. However, polyFAST is not practical for CO₂-EOR field implementation because of its high cost, low yield and environmental constraints [113]. Furthermore, Lemaire and coworkers

recently reported a 19-fold increase in the viscosity of CO₂ upon the addition of 1.5 wt% polyFAST (Mw = 540,000 g/mol) in CO₂ at 15 MPa and 298 K [103]. Generally, fluoropolymers are the most

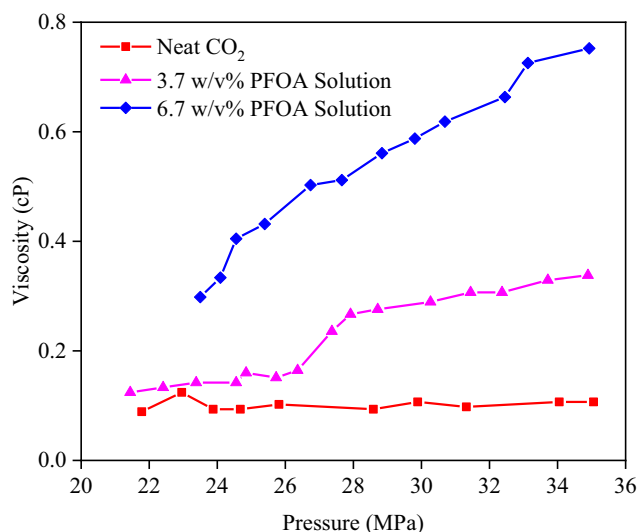


Fig. 5. CO₂ viscosity improvement using PFOA at different experimental conditions [101].

soluble polymers in CO₂ and the most effective CO₂ thickeners than hydrocarbon polymers in the absence of co-solvents [110,114]. However, their field applications are not practical because of their strong adsorption affinity on the rock surface, high costs, and environmental concerns [115].

Siloxane polymers

Siloxane polymers are mainly characterized by silicon-oxygen bonds and have demonstrated remarkable CO₂ thickening performance [55,116–119]. Heller et al. [75] investigated the solubility of high molecular weight polydimethylsiloxane (PDMS, compound 3, Table 1) in CO₂ at 0.03 wt% PDMS, 18.9 MPa and 298 K. However, PDMS did not significantly increase the CO₂ viscosity at this low concentration. Bae and Irani [120] used PDMS (Mw = 197,000 g/mol) to enhance the viscosity of 76% CO₂ using 4 wt% PDMS and 20% toluene at 327 K and 17.2 MPa. Under these conditions, the CO₂ viscosity was increased by 30-fold resulting in delayed CO₂ breakthrough in porous media and the oil recovery was enhanced by 10–20%. In another attempt, Gandomkar et al. [103,121] tested the solubility of PDMS oligomers (4,800 < Mw < 10,000) in CO₂ at 4 wt% PDMS, 298 K and 23.5 MPa, and observed a 3.7 fold CO₂ viscosity increase. Recently, Zhao and co-workers [103,122] have attempted thickening CO₂ with PDMS at 5 wt% PDMS, 5% kerosene, 90% CO₂ and 325 K. Their experimental findings showed that PDMS exhibited a 54-fold increase in CO₂ viscosity. Typically, all previous studies reported that PDMS polymers had higher CO₂-philicity than hydrocarbon polymers [109,123,124]; however, they couldn't viscosify CO₂ without the addition of a significant amount of a co-solvent [56,125,126]. Furthermore, the high cost of PDMS polymers and the large amount of co-solvent needed make the field application of PDMS infeasible.

Hydrocarbon polymers

The polymers in this group primarily consist of carbon and hydrogen atoms only. Currently, poly(1-decene) (P-1-D, compound 4, Table 1) is the only low molecular weight hydrocarbon polymer with high solubility and CO₂ thickening properties [98,127]. Zhang et al. [6] described that less than 1 wt% solution of P-1-D (Mw = 910 g/mol) potentially increased the CO₂ viscosity by 13–14 fold at 329 K. Al Hinai and coworkers [95,101] have recently assessed the feasibility of P-1-D to increase the CO₂ viscosity under reservoir conditions. Their findings revealed that P-1-D was soluble in CO₂ at

higher temperatures and pressure but could not significantly increase the viscosity of CO₂. For example, P-1-D increased the viscosity of CO₂ by 1.2–2.77 fold over the concentration, temperature, and pressure ranges of 0.81–5 wt%, 358–377 K, and 50–55 MPa, respectively [95]. In another attempt, Kar and Firoozabadi investigated the solubility of branched hydrocarbon oligomers of poly(1-decene) in CO₂ and their CO₂ viscosification properties at subsurface conditions. About 1.8 wt% of the investigated oligomers (with 20 repeating units) increased the CO₂ viscosity by 6.5 fold at 308 K and 31 MPa [98]. Recently, Afra et al. [268] evaluated the efficiency of an engineered oligomer of 1-decene to control the mobility of CO₂. Their experimental results demonstrated that the addition of 1.5 wt% of P-1-D (Mw = 2950 g/mol) in supercritical CO₂ at 24 MPa and 363 K has increased the CO₂ viscosity by 4.8 fold.

Oxygenated hydrocarbon polymers

The polymers in this group mainly consist of carbon, hydrogen and oxygen atoms, and they are reported as the most promising CO₂ thickeners than pure hydrocarbon polymers [106]. Several oxygenated hydrocarbon polymers have been designed and investigated to thicken CO₂ under different experimental conditions. Zhang et al. [6] reported that 1 wt% solution of poly(vinyl ethyl ether) (PVEE, Mw = 3800 g/mol, compound 5, Table 1) was able to increase the CO₂ viscosity by 13–14 fold at 329 K. Al Hinai et al. [95] have recently evaluated the CO₂ thickening ability of PVEE under different reservoir conditions. Their findings revealed that PVEE was soluble in CO₂ at higher temperatures and pressure with minor CO₂ viscosifications. For example, PVEE increased the viscosity of CO₂ by 1.2–2.1 fold over the concentration, temperature and pressure range of 1.2–2 wt%, 329–377 K, and 55 MPa, respectively.

In another attempt, Tapriyal [128] formulated a high molecular weight oxygenated hydrocarbon polymer namely poly(vinyl acetate) (PVAc, compound 6, Table 1) to increase the viscosity of CO₂ by dissolving 2 wt% PVAc (MW = 11,000 g/mol) in CO₂ at 64 MPa and 298 K. However, under these experimental conditions, no substantial increase in CO₂ viscosity was noticed. Recently, Lemaire and co-workers [103] also investigated the CO₂ thickening ability of PVAc (MW = 500,000 g/mol) at 2 wt% PVAc, 23 wt% toluene, 75 wt% CO₂, 30 MPa, and 298 K. Their experimental results showed that the addition of a co-solvent (23 wt% toluene) increased the CO₂ viscosity by 70%. Nevertheless, PVAc is the most successful polymeric CO₂ thickener among oxygenated hydrocarbon polymers and the second most CO₂-soluble polymer among non-fluorous polymers after PDMS [129]. However, a large amount of a co-solvent is needed to dissolve PVAc in CO₂ at EOR conditions just like PDMS.

Enick and co-workers [130] have synthesized a new copolymer called poly(benzoyl-vinyl acetate) (PolyBOVA, compound 7, Table 1) to thicken CO₂. Their experimental results demonstrated that the addition of 3.7 wt% PolyBOVA (Mw = 1,400,000 g/mol) in CO₂ at 298 K has increased the CO₂ viscosity by 1.8-fold. Moreover, PolyBOVA is the first non-fluorous polymer with CO₂ thickening ability at low concentration (1 wt%); however, its dissolution in CO₂ requires high pressure (64 MPa).

Recently, Xue et al. [55] have utilized a molecular dynamic simulation method to understand and optimize the chemical properties of poly(vinyl acetate-vinyl ethyl ether) (PVAEE, compound 8, Table 1) as a CO₂ viscosifying agent. The simulation studies showed that the low molecular weight PVAEE (Mw = 4300 g/mol) is capable to increase the CO₂ viscosity by 2–4 fold at a concentration ranging from 1.19–2.35 wt% at 308 K. Although the simulation studies showed that PVAEE could increase the CO₂ viscosity no experimental viscosity data are presented in Xue work to support the molecular dynamic simulation findings.

Al Hinai and coworkers [95] studied the solubility of poly(isobutyl vinyl ether) (Piso-BVE, compound **9**, Table 1) in CO₂ under different experimental conditions. Two concentrations (1.5 and 3 wt%) of Piso-BVE (Mw = 4000 g/mol) were examined at pressures of 50, 53, and 55 MPa and temperatures of 329 to 377 K. The dissolution of 1.5 wt% Piso-BVE in CO₂ at 329–377 K resulted in a 1–1.23 fold increase in CO₂ viscosity. Nonetheless, the steric effect and an increase in the alkyl side chain length of Piso-BVE have been reported to affect negatively the CO₂ thickening ability of the polymer.

Bullen et al. [131] have published a patent that claimed that poly(ether carbonate) (PEC, Mw = 20,000–150,000 g/mol, compound **10**, Table 1) exhibits dissolution in CO₂. Their findings showed that PEC increased the CO₂ viscosity by 3 fold at a concentration, temperature, and pressure of 2.5 wt%, 295 K, and 10–25 MPa, respectively. Moreover, Sarbu et al. [132] also investigated the solubility of poly(ether carbonate) (Mw = 16000 g/mol) in CO₂. However, they found that poly(ether carbonate) could only dissolve in CO₂ at a concentration, temperature, and pressure of 1 wt%, 295 K, and 14 MPa, respectively, without a significant increase in CO₂ viscosity.

Zhang et al. [133] reported that low molecular weight poly(propylene oxide) (PPO, compound **11**, Table 1) showed high solubility in dense CO₂. When 1 wt% PPO (Mw = 409 g/mol) was dissolved in CO₂ at 306 K and 17.9 MPa; the CO₂ viscosity was increased by 1.25 fold [114,133]. Although PPO is the most soluble polymer in CO₂ among low molecular weight polymers, no substantial increase in CO₂ viscosity was detected. The high solubility of PPO in CO₂ is ascribed to the weaker PPO–PPO self-association and stronger CO₂–PPO interactions [114,134].

Shen et al. [109] have studied the solubility of 5 wt% poly(methyl acrylate) (PMA, compound **12**, Table 1) oligomers in CO₂ at different experimental conditions. PMA oligomer (Mw = 1390 g/mol) was found to dissolve in CO₂ at 34.6 MPa and 298 K. Shen also reported that PMA oligomer (Mw = 2848 g/mol) could dissolve in CO₂ at 298 K and 89.1 MPa without a significant increase in CO₂ viscosity.

Enick and co-workers [135] have formulated poly(propylene glycol) (PPG, compound **13**, Table 1) as a CO₂-philic compound to replace the fluoroalkyl tails in fluorinated polymers. They reported that PPG has strong Lewis acid–Lewis base interactions with CO₂ and has attracted researchers' attention because of its low chain flexibility and high solubility in CO₂. Hong and co-workers also reported that low molecular weight PPG (Mw < 1000 g/mol) is CO₂ soluble; however, PPG becomes CO₂ insoluble as the molecular weight increases (Mw > 2000 g/mol) [134]. Although low molecular weight PPG was more CO₂ soluble, no CO₂ viscosification data were reported; thus, the CO₂ thickening ability of PPG needs to be evaluated.

Conway et al. [136] have examined the solubility of poly(lactic acid) (PLA, compound **14**, Table 1) in neat CO₂. Their experimental results demonstrated that PLA (Mw = 128,500 g/mol) dissolves in CO₂ at a high concentration (5 wt%) and pressure (140 MPa), and a moderate temperature (308 K). The copolymers of PLA are insoluble in CO₂ at low pressure; thus, they are not suitable CO₂ thickeners [106,109].

Shen et al. [109] studied the solubility of poly(vinyl formate) (PVF, compound **15**, Table 1) in CO₂ at 484 K and 240 MPa. Their findings revealed that PVF is insoluble in CO₂; these results are as per the previously reported one by Rindfleisch and coworkers [57]. Although the formate hydrogen (proton) is not very acidic, it facilitates stronger PVF self-interactions than CO₂–PVF interactions; thus, making PVF CO₂ insoluble [109].

Shi et al. [137] investigated the CO₂ thickening ability of poly(urethane disulfates) (PUD, Mw = 32,500 g/mol, structure not reported) using falling cylinder viscometry at 4 wt% PUD, 298 K

and 34.5 MPa. The addition of PUD in CO₂ increased the solution viscosity by 2.7-fold relative to neat CO₂ [73]. Higher molecular weight PUD is crucial to dramatically increase the viscosity of CO₂ through non-covalent interaction when the polymer is present in dilute concentration.

Nitrogen-based polymers

The solubility and CO₂ thickening properties of nitrogen-containing polymers such as poly(vinyl amine), poly(2-ethyl-2-oxazoline), poly(propyleneimine), poly(acrylamide), poly(propylmethyl acrylateethylamine), poly(4-vinyl pyridine), poly(2-vinyl pyridine), and poly(N-vinyl imidazole) have been previously examined [106,138]. However, none of these polymers was observed to be soluble in CO₂ at dilute concentrations (below 0.7 wt% polymer), and temperature and pressure of up to 343 K and 55 MPa, respectively. Enick and co-workers [106] reported the poor solubility of poly(4-vinyl pyridine) (P4VP, compound **16**, Table 1) in CO₂ which is attributed to the stronger polymer–polymer interactions than polymer–CO₂ interactions confirmed through molecular modelling calculations and experimental results.

In summary, several polymeric CO₂ thickeners have shown promising performance in increasing the viscosity of CO₂. Different parameters such as pressure, temperature, the molecular weight of the polymer, presence or absence of co-solvents, the solubility of the polymers in CO₂, concentration/amount of polymers dissolved in CO₂, crude oil composition and reservoir properties (porosity, permeability, salinity, rock type, etc.) are usually considered in evaluating the performance of polymeric CO₂ thickeners [37,139]. For field implementations, other factors such as the thickening ability of the polymers, the amount of oil recovered, economic issues and environmental concerns are also taken into consideration. The important parameters used in evaluating the performance of the polymeric CO₂ thickeners reviewed in this paper are summarized in Table 2.

Polymeric CO₂ thickening mechanism

The solubility and CO₂ thickening properties of polymers are determined mainly by polymer–CO₂ interactions [103]. The stronger the polymer–CO₂ interaction or the weaker the polymer–polymer interaction, the more likely the polymer to dissolve and thicken the CO₂ solution [105,141]. Polymers usually tend to thicken CO₂ through polymer coil expansion, intermolecular interactions, entanglement, aggregation, and self-assembly [101,142–144]. The polymers with CO₂-philic or Lewis acid–base functionalities such as carbonyls, ethers, acetates, amide, and esters are likely to dissolve in CO₂; however, the presence of hydroxyl (OH) and carboxylic acid (COOH) groups in the polymer chain make the polymer more CO₂-phobic and impart insolubility in dense CO₂ [101,145–147]. Moreover, polymers with relatively small alkyl chains and highly branched alkyl chains are reported to be more CO₂-philic than polymers with longer and linear alkyl groups [103,134]. The Lewis base functional groups in polymers act as an electron donor to electron-deficient carbon in CO₂ (Lewis acid) and facilitates Lewis acid–Lewis base (La–Lb) interactions which enhance the CO₂ viscosity [102,106]. Furthermore, the electronegative oxygen atoms in CO₂ have been reported to promote weak hydrogen bonding (hb) interactions with the hydrogens on the polymer backbone or side chain [50,101,106,116]. The intermolecular interactions between the polymers and CO₂ create a three-dimensional network which restricts the free movement of the molecules in a solution leading to the formation of viscosity-enhancing supramolecular structure in solution [70,148]. In this study, the La–Lb and hb interactions between the polymer and CO₂ are described using poly(vinyl amine) as an example (Fig. 6).

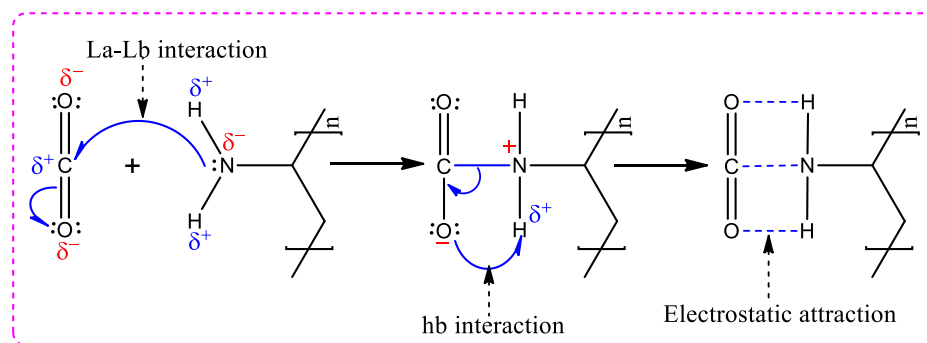


Fig. 6. Intermolecular interactions between polymer and CO₂.

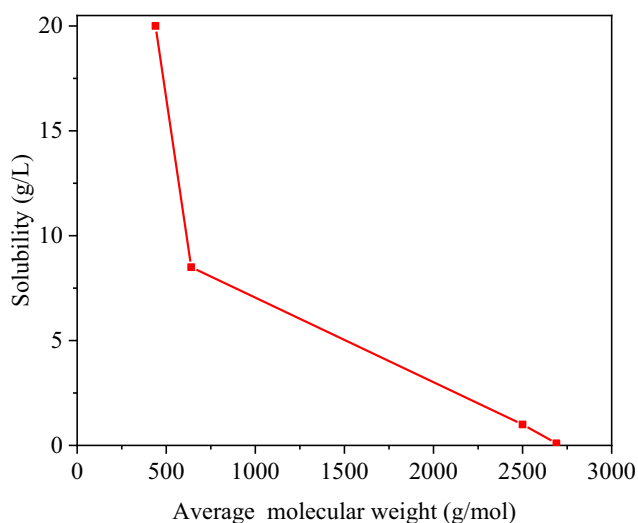


Fig. 7. Effect of molecular weight on poly(butene) solubility in CO₂ at 11.7 MPa and 298 K. Reproduced with permission from ref. [75] Copyright 1985 Society of Petroleum Engineers.

Factors affecting polymeric CO₂ thickeners

The major factor facing polymeric CO₂ thickeners at a laboratory scale is the solubility of polymers in CO₂ [95]. The solubility and CO₂ thickening abilities of the polymers are influenced by several polymer properties such as stereochemistry and molecular weight of the polymers [75]. The presence of CO₂-philic groups or aliphatic side chains/length and their arrangement in the polymer backbone can either enhance or reduce the CO₂ viscosity

Table 3

The summary of studies on small molecule CO₂ thickeners discussed in this paper.

Compound	Concentration of thickener (wt%)	Co-solvent	Experimental condition	Solubility in CO ₂	CO ₂ thickening ability	Reference
BBT	1.6	Hexane 48.4 wt%	298 K and 8.8 MPa	Soluble	300 fold	[78]
12-HSA	3	Ethanol 15 wt%	307 K and 12.4 MPa	Soluble	100 fold	[161]
FBU	5	No co-solvent	298 K and 31 MPa	Soluble	3–5 fold	[137]
NFBU	1	No co-solvent	298 K and 62 MPa	Soluble	Not Significant	[162]
TATF	0.13	Pentane 39 wt%	297 K and 6.9 MPa	Insoluble	No viscosity increase	[84,101]
SFTATF & FTI	2–4	No co-solvent	298 K and 16.5 MPa	Soluble	2–3 fold	[140]
HAD2EH	2	Hexane 23 wt%	298 K and 34.5 MPa	Insoluble	No viscosity increase	[103]
CPE	-	Hexane	-	Soluble	Not Significant	[103]

[149]. The polymer pendant groups are usually arranged in an isotactic, syndiotactic or atactic manner [75]. The atactic polymers are amorphous due to the high degree of irregularity in their structures and are more CO₂ soluble than isotactic and syndiotactic polymers. In the case of polymer molecular weight, high molecular weight polymers are reported to have low solubility in CO₂ while low molecular weight polymers are highly soluble in CO₂ inducing minor viscosification. Heller and co-workers [75] have studied one sequence of polybutene with different molecular weights in understanding the effect of polymer molecular weight on their solubility in liquid CO₂. The results obtained indicated that the solubility of polybutene in CO₂ decreases with increasing polybutene molecular weight (i.e., 440, 640 and 2,500 g/mol induced a solubility of 20, 8.5 and 1.5 g/L in CO₂, respectively) at 298 K and 11.7 MPa as shown in Fig. 7. Generally, the solubility of polymers in CO₂ decreases with increasing polymers' molecular weight; however, an average molecular weight (6,000–15,000 g/mol) has shown promising solubility and CO₂ thickening properties [75]. Therefore, when developing polymeric CO₂ thickeners; one should critically consider the stereochemistry and molecular weight of the polymers.

Small molecule CO₂ thickeners

The application of small self-interacting molecules is an alternative method to polymers for direct CO₂ thickening [101,103,150]. This class of compounds contain at least one CO₂-philic group and one CO₂-phobic group [50,70]. The CO₂-philic groups promote the dissolution of the compound in CO₂ while the CO₂-phobic groups interact/associate with the CO₂-phobic groups of the neighbouring molecules [50,151,152]. However, previous literature

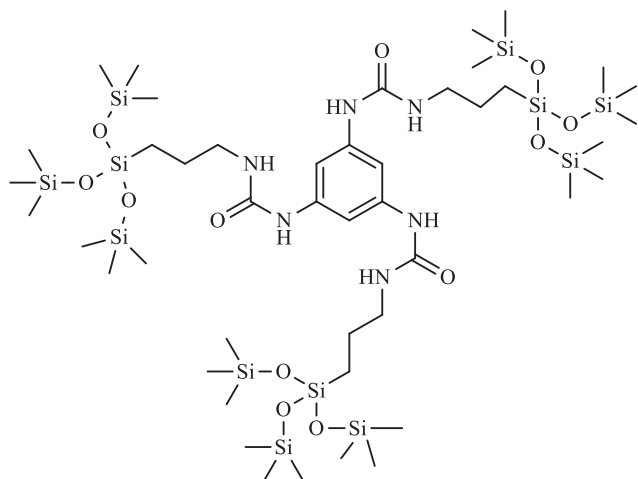


Fig. 8. Molecular structure of benzene trisurea as small molecule CO₂ thickener. Reproduced with permission from ref. [151] Copyright 2017 University of Pittsburgh.

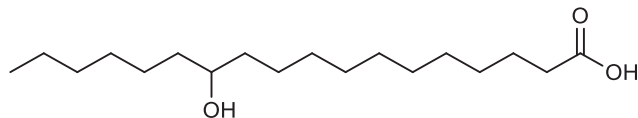


Fig. 9. Molecular structure of 12-hydroxystearic acid. Reproduced with permission from ref. [103] Copyright 2021 American Chemical Society.

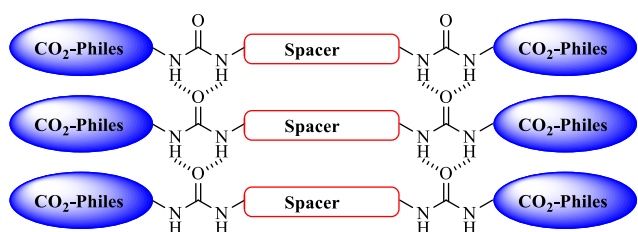


Fig. 10. Intermolecular hydrogen bonding of non-fluorinated bisureas containing CO₂-philic groups. Reproduced with permission from ref. [162] Copyright 2007 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

reports that if one chooses a thickener with the extremely powerful associating group then the thickener might turn CO₂-insoluble, but if the extremely weak associating group is integrated, the thickener may dissolve in CO₂ without a substantial increase in CO₂ viscosity [103,153]. Ideally, it's reported that a good CO₂ thickener must be soluble enough in CO₂ at reservoir conditions without the need for co-solvents and must increase the CO₂ viscosity by a factor of 2–100 fold in a concentration of less than 1wt% [52,154]. The unique molecular structures of small molecule thickeners generally promote inter-molecular associations in solution and form a viscosity-enhancing macromolecular network [70]. However, the CO₂ viscosity-enhancing ability of small molecule thickeners decreases as the intermolecular attraction forces that favour self-assembly are destroyed at a higher temperature [70]. Moreover, the association of small molecules in solution can be described through remarkable changes in CO₂ solution viscosity. However, techniques such as scanning electron microscope (SEM), small-angle neutron scattering (SANS), X-ray diffraction (XRD) and differential scanning calorimetry (DSC) can also be used to confirm such associations [155–160].

Currently, several attempts have been conducted by different researchers to thicken CO₂ using small molecule compounds and

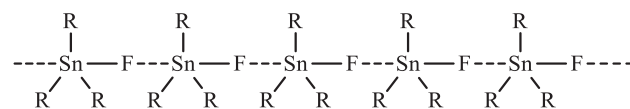


Fig. 11. Association mechanism between trialkyltin fluoride molecules. Reproduced with permission from ref. [165] Copyright 2016 Society of Petroleum Engineers.

their research findings are summarized in Table 3. The most commonly investigated small molecule CO₂ thickeners include branched benzene trisurea (BBT), 12-hydroxystearic acid (12-HSA), fluorinated bisureas (FBU) and non-fluorinated bis-ureas (NFBU), trialkyltin fluoride (TATF), semi-fluorinated trialkyltin fluorides (SFTATF) and fluorinated telechelic ionomers (FTI), hydroxylaluminum di(2-ethyl hexanoate) (HAD2EH) and cross-linked phosphate esters (CPE) as presented in Table 3. The descriptions of the research findings summarized in Table 3 are given in Sections 3.2.1 to 3.2.6.

Branched benzene trisurea

Recently, Doherty et al. [78] have synthesized and investigated several BBT compounds to develop non-fluorous small molecule CO₂ thickeners. The designed compounds contain cyclic or aromatic core rings in their molecular structure as CO₂-phobic segments (Fig. 8). The aromatic or cyclic systems are incorporated with the CO₂-philic groups such as ether, amide, ester or urea to demonstrate the intermolecular interaction which is essential for CO₂ viscosity enhancement. Doherty's research findings revealed that branched benzene trisurea (1.6 wt%) is soluble in dense CO₂ with a remarkable viscosity increase (300-fold) in the presence of hexane (48.4 wt%) at 298 K and 8.8 MPa [78,104]. This extreme viscosity increase and large amount of co-solvent used limits the application of benzene trisurea in EOR field applications.

12-Hydroxystearic acid

Heller and colleagues [161] had previously investigated 12-HSA (Fig. 9) to gel light hydrocarbon, halogenated solvents and CO₂. However, their experimental results revealed that 12-HSA is not soluble in CO₂ unless a substantial amount of ethanol is added as a co-solvent. The addition of 15 wt% ethanol and 3 wt% 12-HSA in CO₂ resulted in an increased CO₂ viscosity by 100-fold at a pressure and temperature of 12.4 MPa and 307 K, respectively.

Fluorinated and non-fluorinated bisureas

A team of researchers at the University of Pittsburgh and Yale University formulated small molecule compounds comprising one or two urea groups [137]. The urea groups in the synthesized compounds are reported to cause self-assembly interactions through hydrogen bonding (Fig. 10) that can increase the viscosity of CO₂-rich solutions. Their research findings demonstrated that out of 12 examined FBU compounds, only 4 compounds were extremely soluble in CO₂ without heating and were able to increase the CO₂ viscosity by 3–5 times at 5 wt% FBU, 31 MPa and 298 K. In another attempt Paik and co-workers designed NFBU compounds by incorporating the CO₂-philic groups into the molecular structure of the bisurea compounds as shown in Fig. 10 [162]. However, their findings showed that the designed compounds underwent self-assembly and precipitated out of solutions without any significant increase in CO₂ viscosity.

Trialkyltin fluorides

A series of TATF compounds were investigated by Heller et al. [163] as CO₂ thickeners. Tributyltin fluoride (TBTF) molecule is an example of a TATF compound that showed a remarkable increase in CO₂ viscosity via intermolecular associations between

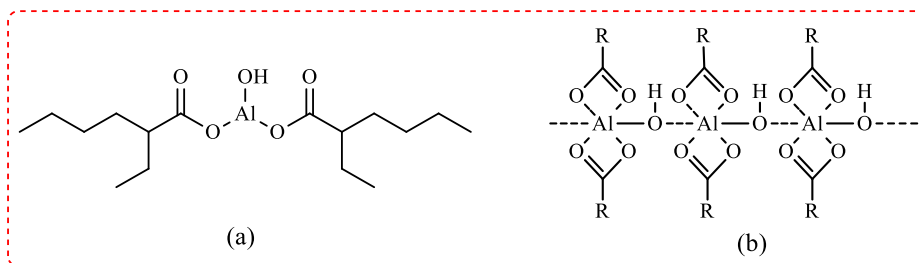


Fig. 12. Molecular structure of (a) HAD2EH and (b) its possible association mechanism. a) Reproduced with permission from ref. [103] Copyright 2021 American Chemical Society. b) Reproduced with permission from ref. [165] Copyright 2016 Society of Petroleum Engineers.

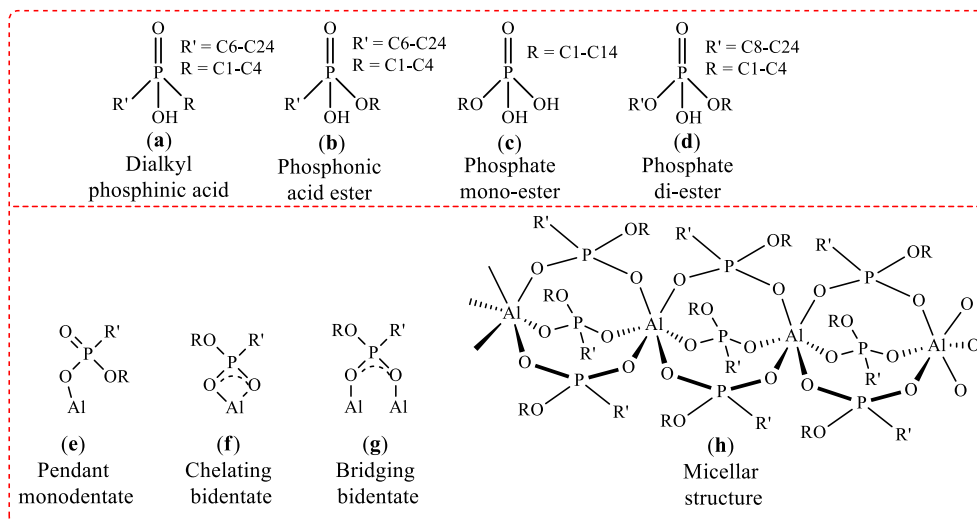


Fig. 13. Molecular structure of phosphorus-based esters (a-d) and polyvalent metal ion interactions (e-h). Reproduced with permission from ref. [165] Copyright 2016 Society of Petroleum Engineers.

Table 4
Classification of surfactants based on their chemical structures and charges [178].

Type	Molecular structure	Example
Cationic		
Anionic		
Nonionic		
Amphoteric		

tin (Sn) and fluorine (F) atoms of the neighbouring molecules in the solution [50,163]. The tin atom is fairly electropositive; thus, it interacts with the fluorine atom which is electronegative to form Sn-F association (Fig. 11) whereas the alkyl arms in TATF create a free volume which facilitates the solubility of TATF in CO₂ [140]. The investigated TATF compounds were found insoluble in CO₂ with minor viscosifications even in the presence of a co-solvent. However, they were successful in thickening light hydro-

carbons such as propane, butane, pentane and hexane [84,163,164]. Shi and co-workers [140] afterwards synthesized SFTATF and FTI to enhance the solubility of TATF in CO₂. Their findings showed that 2–4 wt% tri(2-perfluorobutyl ethyl) tin fluoride was able to increase the CO₂ viscosity by 2–3 fold at 298 K and 16.5 MPa without the addition of a co-solvent. Generally, TATF and their ionomers are not regarded as viable CO₂ thickeners due to their high costs and environmental concerns [50,70].

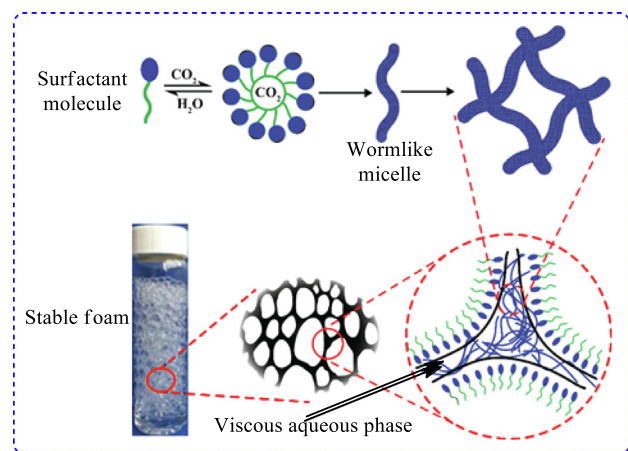


Fig. 14. Surfactant molecules self-assembled into wormlike micelles forming stable CO₂ foam. Reproduced with permission from ref. [188] Copyright 2018 Elsevier.

Hydroxyaluminum disoaps

The thickening abilities of HAD2EH (Fig. 12a) were initially investigated in light hydrocarbons (propane, butane, pentane and hexane) by different researchers [103,166]. For example, the addition of 0.2–1.0 wt% HAD2EH at 293 K has increased the viscosity of compressed liquid propane and butane by a factor of 10–100 fold [165]. Currently, Lemaire and co-workers have investigated the CO₂ thickening ability of 2 wt% HAD2EH in 75 wt% CO₂ at 298 K and 34.5 MPa in the presence of 23 wt% hexane [103]. The HAD2EH disoaps were found to promote inter-molecular associations in CO₂ solution and form a viscosity-enhancing macromolecular network (Fig. 12b). Lemaire research findings indicated that HAD2EH is insoluble in dense CO₂ at 298 K, its solubility may be increased through the addition of a co-solvent such as alcohol or carboxylic acid; however, this has not yet been examined.

Cross-linked phosphate esters (CPE)

CPE compounds were initially utilized to thicken a variety of light alkanes during hydraulic fracturing in water-sensitive formations [167–170]. The CPE groups (Fig. 13a–d) were reported to coordinate with the polyvalent metal ions (Al³⁺, Fe³⁺, Mg²⁺, Ti⁴⁺ and Zn²⁺) in the hydrocarbon phase forming metal complexes (Fig. 13e–g) and a web-like supramolecular network structure (Fig. 13h) that increases the solution viscosity [165,171–173]. Lemaire and colleagues [103] have currently investigated the CO₂ thickening abilities of commercially available CPEs. None of the investigated CPEs was found to thicken the CO₂-rich solution despite their high solubility in CO₂. Thus, they suggested that to thicken a CO₂-rich solution with CPE, highly CO₂-philic groups such as sugar acetates, oligovinyl acetate or oligomers of propylene oxide need to be incorporated in the phosphate ester structures and a high concentration of a co-solvent (50% hexane) must be used [103].

Surfactant CO₂ thickeners

Surfactants are commonly utilized as CO₂ foam stabilizing agents in CO₂ flooding during EOR [174–176]. A surfactant is an active chemical agent that lowers the IFT of a multiphase system in which it's dissolved [177]. Structurally, surfactants are classified as amphipathic organic compounds, meaning that their molecules have a long hydrophobic group and a small hydrophilic group [178]. However, they are further classified into cationic, anionic, nonionic and amphoteric surfactants based on their charges [179–181] (Table 4).

Surfactants control CO₂ mobility mainly through wettability alteration and interfacial tension reduction [182]. The surfactant molecules form micelles around CO₂ molecules in aqueous solutions leading to stabilized CO₂ foam and increased solution viscosity [183–185]. They also self-assemble into long wormlike structures and subsequently entangle with one another into network structures forming a CO₂ viscous solution with stable foam [181,186,187] (Fig. 14).

Several attempts have been made in identifying appropriate surfactants for CO₂ mobility control in EOR processes [189–192]. A lot of literature has reported on CO₂ mobility and conformance control (uniformity of flood front of the injected drive fluid) using surfactants; however, there are limited numerical data on CO₂ viscosification by surfactants. Eastoe and co-workers have attempted to increase the viscosity of CO₂ by using commercially available surfactants [193]. Nevertheless, none of the commercially available surfactants was found to be soluble in CO₂. To overcome the challenges associated with surfactant solubility in CO₂, commercially available surfactants have been modified by incorporating CO₂-philic functional groups [194]. Various surfactants have been designed and investigated for CO₂ mobility control as presented in Table 5. In this study, the surfactants presented in Table 5 are categorized as fluorinated, amine and oxygenated-based surfactants as described in Sections 3.3.1 to 3.3.3.

Fluorinated surfactants

A series of fluorinated and semi-fluorinated surfactants (compounds 1–5, Table 5) have been designed and investigated for CO₂ mobility control [196,198,204–206]. Both fluorinated and semi-fluorinated surfactants were found CO₂ soluble and increased the CO₂ viscosity by forming rod-like micelles in the presence of a small amount of water [207]. Eastoe et al. [196] have reported a significant increase in CO₂ viscosity from sodium pentadecfluoro-5-dodecyl sulphate (NaF7H4) and nickel bis-nonofluoropentane sulphosuccinate (Ni-diHCF4). A CO₂ viscosity improvement of up to 1.5 fold was noticed at 298 K, 35 MPa and 6 wt% Ni-diHCF4 upon the addition of 10 moles of water whereas at 313 K, 40 MPa and 4.4 wt% NaF7H4, a CO₂ viscosity increase of up to 2 fold was observed in the presence of 12.5 moles of water [195,196]. Moreover, these thickeners are not suitable for direct field applications in EOR because high pressure and concentration are required to achieve a small increase in CO₂ viscosity.

Amine surfactants

Amine surfactants (compound 6–12, Table 5) have been studied as CO₂ foaming agents at high temperatures and high salinity conditions [199,200,208]. A mixture of 1.0 wt% (cocamidopropyl dimethylamine, oleamidopropyl dimethylamine and erucamidopropyl dimethylamine) surfactant and 20,000 mg/L brine water was saturated with CO₂ and the viscosity of the bulk solution was measured at 403 K and 10.5 MPa [199]. The experimental findings revealed that the investigated amine surfactants had increased the viscosity of the bulk solution through self-assembling behaviours and the formation of three-dimensional networks. Furthermore, the stability of amine surfactants and their CO₂ viscosification ability increases as the carbon chain length increases.

Oxygenated surfactants

Several oxygenated hydrocarbon surfactants (compound 13–22, Table 5) have been formulated and used to generate CO₂-stable foams [183,202]. Oxygenated hydrocarbon surfactants have shown promising CO₂ mobility control and exhibited high solubility in CO₂ to a level similar to those attained by fluorinated ionic surfactants [202]. The oxygenated hydrocarbon surfactants with sugar acetate functionalities such as oligo(vinyl acetate) were also deter-

Table 5
Molecular structures of CO₂-philic surfactants reviewed in this work.

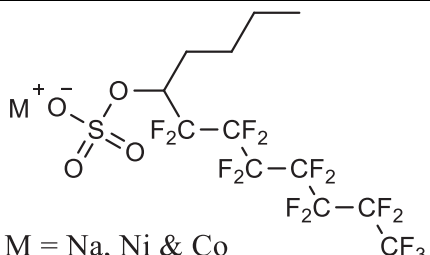
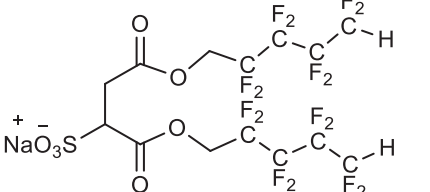
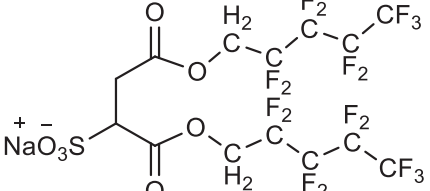
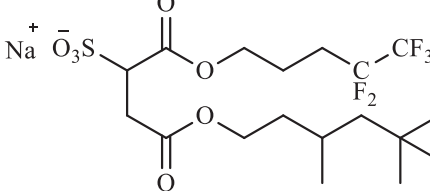
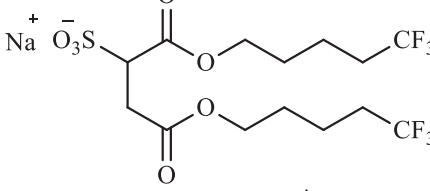
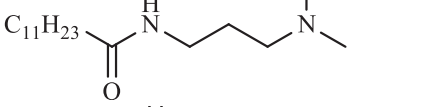
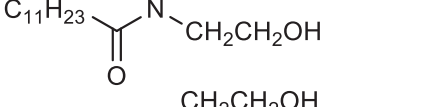
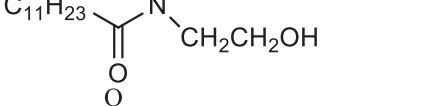
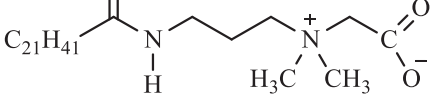

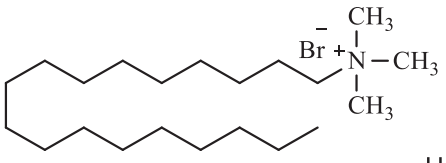
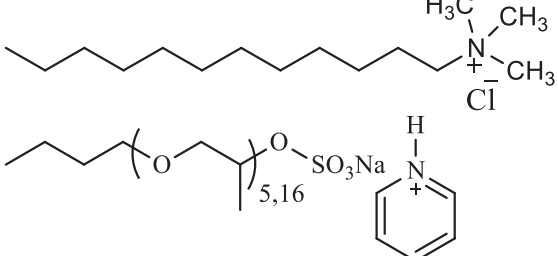
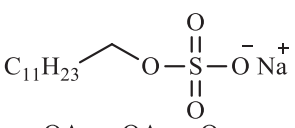
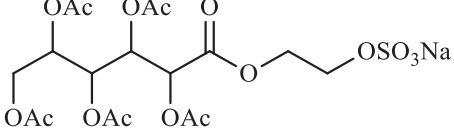
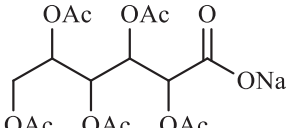
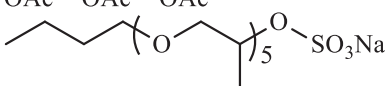
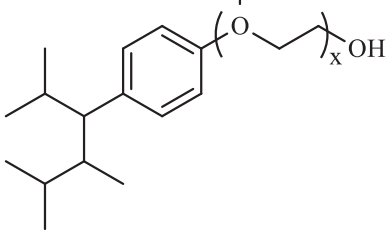
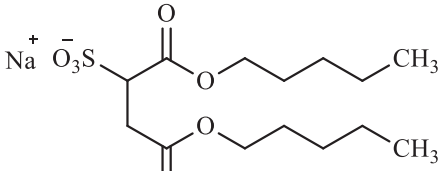
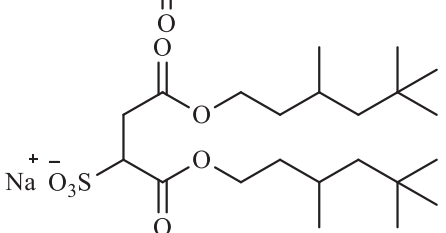
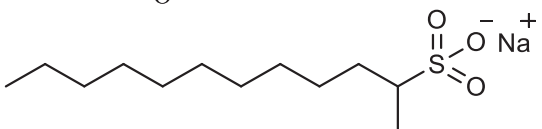

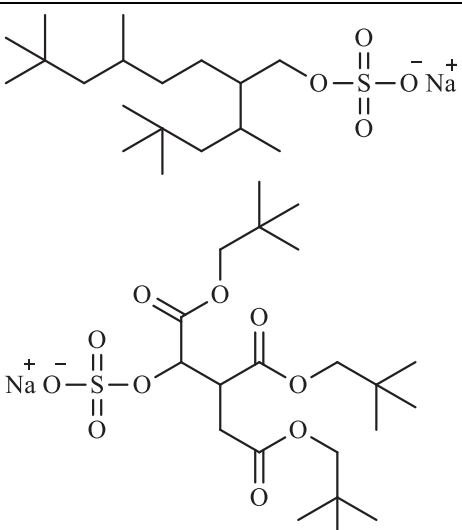
Compound	Molecular structure	Surfactant name	Reference
1	 <p>M = Na, Ni & Co</p>	Pentadecafluoro-5-dodecyl (M-F7H4)	[195]
2		Sodium bis(1H,1H,5H-octafluoropentyl)-2-sulfosuccinate (Na(di-HCF4))	[196]
3		Sodium bis(1H,1H-perfluoropentyl)-2-sulfosuccinate (Na(di-CF4))	[197]
4		Sodium (4H,4H,5H,5H,5H-pentafluoropentyl)-3,5,5-trimethyl-1-hexyl)-2-sulfosuccinate (Hybrid CF2/AOT4)	[183,197]
5		Sodium bis(5H,5H,5H-trifluoropentyl)-2-sulfosuccinate (Di-CF1)	[183,198]
6		Cocamido-propyl dimethyl amine (UC11AMP)	[199,200]
7		Coconut monoethanol amide (CMEA)	[200]
8		Lauryl diethanol amide (LDEA)	[200]
9		Erucamidopropyldimethylamine (EA)	[192,201]
10		N,N,N-trimethyloctadecan-1-aminium	[185]

Table 5 (continued)

Compound	Molecular structure	Surfactant name	Reference
11		Arquad 12-37 W	[74]
12		Pyridinium sulfate (PPGMBE)	[202]
13		Sodium dodecyl sulfate (SDS)	[199,200]
14		Peracetyl gluconic ethyl sodium sulfate	[202]
15		Peracetyl gluconic sodium carboxylate	[202]
16		Sodium sulfate PPGMBE	[202]
17		Nonylphenol ethoxylate	[176,190]
18		Sodium bis(pentyl)-2-sulfosuccinate (di-C5SS)	[183,198]
19		Sodium bis(3,5,5-trimethyl-1-hexyl)-2-sulfosuccinate (AOT4)	[203]
20		Alpha-olefin sulfonate (AOS)	[181]
21		Sodium 2-(4,4-dimethylpentan-2-yl)-5,7,7-trimethyloctylsulfate (SIS1)	[183]

(continued on next page)

Table 5 (continued)

Compound	Molecular structure	Surfactant name	Reference
22		Sodium 1,4-bis(neopentylloxy)-3-(neopentylloxycarbonyl)-1,4-dioxobutane-2-sulfonate (TC14)	[183]

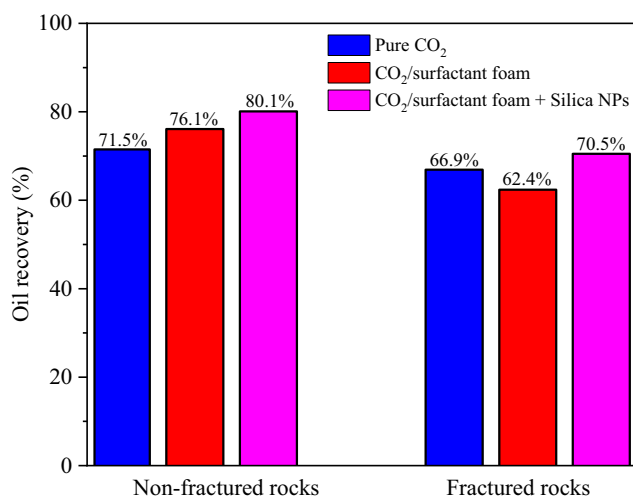


Fig. 15. Effect of silica nanoparticles on oil recovery of Bentheimer sandstone cores. Reproduced with permission from ref. [231] Copyright 2020 Springer.

mined to be CO₂ soluble by 2–7 wt% [202]. The oxygen atoms on the surfactant molecule make the surfactant more CO₂-philic and substantially improve the mobility of CO₂ during EOR operations [37].

Despite surfactants being extensively used as foaming agents in CO₂-flooding, surfactant-stabilized CO₂ foams are thermodynamically unstable; they tend to degrade and precipitate under harsh reservoir conditions [200,201,209]. Anionic and nonionic surfactants are the most CO₂ soluble foaming agents with greater thermal stability and CO₂ mobility reduction at a low operational cost [181,210–213]. However, amphoteric surfactants perform much better in terms of CO₂-foam stability as compared to nonionic and anionic surfactants [214]. Cationic surfactants are not stable at high temperatures and salinity reservoir conditions [199,211,215]. The majority of fluorinated surfactants are costly, environmentally unfriendly and tend to adsorb on the porous

reservoir rocks; thus, they need to be added in large quantities to compensate for the loss resulting from adsorption [74,191]. Anionic surfactants are often utilized used foaming agents in EOR field applications because they are commercially available, less expensive, have good foaming properties and have a high tolerance to temperature and salinity [214,216,217]. Therefore, it is essential to design appropriate surfactants with excellent properties for CO₂ mobility control in EOR.

Nanoparticle CO₂ thickeners

Nanoparticles have gained tremendous interest in the oil and gas industry for being used as additives in CO₂ flooding, drilling fluids and oil well cement [218,219]. Nanoparticles are small particles of matter whose diameter ranges between 1 to 100 nanometers (nm) in size [80,218,220]. A variety of nanoparticles (NPs) such as Silver (Ag), Silicon dioxide (SiO₂), palladium (Pd), Copper(II) oxide (CuO), Iron(III) oxide (Fe₃O₄), Titanium dioxide (TiO₂), Nickel oxide (NiO), Aluminum oxide (Al₂O₃), Cobalt(III) oxide (Co₃O₄), boron nitride (BN) and graphene oxide (GO) nanoparticles have been investigated to generate a stable CO₂ foam at a laboratory scale [37,221–228]. Among investigated nanoparticles, SiO₂ nanoparticles have demonstrated superior performance in terms of CO₂ foam stability and gas mobility control because of their ability to remain dispersed uniformly in aqueous solutions [37,229,230]. For example, Al Yousef et al. [231] have reported a positive effect on oil recovery upon utilization of surface-modified silica nanoparticles to control CO₂ mobility and enhance oil recovery (Fig. 15).

The application of nanoparticles for CO₂-foam stability is an alternative method to surfactant in EOR operations under harsh reservoir conditions such as high temperatures, salinity, shear and pressures [229,231–233]. Nanoparticles are reported to enhance the performance of CO₂-EOR through wettability alteration, pore channels plugging, interfacial tension (IFT) reduction and CO₂ viscosity increase [230,234,235]. The dissolution and dispersion of nanoparticles in CO₂ for CO₂-foam stability have been investigated by several researchers [223–227]. However, there is

a limited number of studies on the use of nanoparticles as direct CO₂ thickeners. Shah and Rusheet [236] have attempted to thicken CO₂ having the initial viscosity of 0.016 cP with 1wt% copper oxide (CuO) nanoparticle in the presence of 5 wt% PDMS at 323 K and 17.2 MPa. Their research findings showed that the viscosity of thickened CO₂ by CuO is 140 times greater than that of conventional CO₂.

Recently, Gandomkar and Sharif [222] investigated the CO₂ thickening ability of nanocomposite (P-1-D and graphene oxide nanoparticle) at 353 K and 19.8 MPa. Their experimental findings demonstrated that the addition of P-1-D and graphene oxide enhanced the CO₂ viscosity by 23-fold without the use of a co-solvent. In another attempt, Zhang and colleagues [237] designed a CO₂ thickener by combining copolymers (partially sulfonated styrene and fluorinated polymers) with nanoparticles. Their experimental results indicated that in the presence of 1 wt% nanocomposite fibre, the designed copolymers increased the viscosity of CO₂ by 100-fold compared to net CO₂ at 333 K and 28 MPa. Recently, the rheological properties of supercritical CO₂ containing different nanoparticles (CuO and Al₂O₃) have been studied using multi-scale computational and simulation methods [238–240]. The multi-scale computational modelling study using 1 vol% CuO nanoparticles dispersed in CO₂ predicted the CO₂ viscosity increase of 30–150% [238]. The application of nanoparticles in CO₂-EOR to thicken CO₂ is in its youngest stage and primarily validated only at a small laboratory scale. Therefore, more research (both experimental, simulation and actual field tests) needs to be conducted to disclose their CO₂ thickening ability and feasibility for field applications to enhance oil recovery.

Comparison between CO₂ thickening technologies

The CO₂ thickening methods which are commonly investigated include the use of polymers, surfactants, small molecule compounds and nanoparticles. The suitability of these methods for EOR applications mainly depends on the solubility of the thickener in CO₂, the cost of the thickener, environmental concerns, thick-

ener performance under harsh reservoir conditions (high salinity, temperatures and pressures) and the amount of co-solvent required to attain higher CO₂ viscosity. Based on these factors, the working principle, advantages and disadvantages of different CO₂ thickening technologies are compared as presented in Table 6.

Field-scale application of CO₂ thickeners

Although there is an extensive history of attempts to viscosify CO₂ using polymers, surfactants, small molecule compounds or nanoparticles; most of the attempts are based on laboratory or simulation studies with a limited number of field-scale tests [38]. Several pilot field tests have been reported on the use of polymers and surfactants for CO₂ mobility and conformance control during EOR as summarized in Table 7. However, currently, no pilot field tests have been reported on the use of small molecule and nanoparticle CO₂ thickeners to improve the performance of CO₂ flooding. The research findings summarized in Table 7 are described in the subsequent paragraphs

Hild and Wackowski [241] have reported on the use of polyacrylamide gel to improve CO₂ flooding performance at Rangely Weber Sand Unit in north-western Colorado, USA. A substantial amount (10,000 bbls) of polymer gel has been injected into 44 wells. The results obtained from 44 injection wells showed higher oil output of 21 bbls/day and a substantial reduction in CO₂ mobility. Karaoguz et al. [242,251] reported another successful pilot field implementation of cross-linked polyacrylamide gel for CO₂ conformance control at the Bati Raman heavy-oil field in South-eastern Turkey. A huge amount (6500 11,000 bbls) of polymer gel was injected into 3 wells. This attempt was technically and economically successful, the rate of oil production was increased from 645 stb/day to 720 stb/day after the polymer gel treatment. Lantz and Muniz [243] have reported on the usage of a partially hydrolyzed polyacrylamide (PHPA) gel to control CO₂ conformance in the SACROC unit, in Snyder County, Texas, USA. The results obtained from the gel treatment showed an incremental oil output

Table 6
Comparison between CO₂ thickening technologies reviewed in this work.

Method	Working principle	Advantages	Disadvantages
Polymers	Polymer coil expansion, intermolecular interactions, entanglement and self-assembly.	<ul style="list-style-type: none"> -Increase the CO₂ viscosity over a wide range of temperatures. -Fluorinated polymers thicken CO₂ without the addition of co-solvents. -Mostly investigated and have shown excellent performance over other methods. 	<ul style="list-style-type: none"> -High molecular weight polymers hardly dissolve in dense CO₂. -Some polymers require co-solvents to dissolve in CO₂ and are costly. -Polymer solubility in CO₂ mostly occurs above supercritical conditions. • Fluorinated polymers exhibit strong adsorption affinity on the rock surface.
Small molecule compounds	Intermolecular associations forming viscosity-enhancing macromolecular networks in solution.	<ul style="list-style-type: none"> -Low concentration can induce a significant increase in CO₂ viscosity. -Small molecules easily gel the bulk fluids leading to a more viscous solution. -Forms stable single-phase viscous solution. 	<ul style="list-style-type: none"> -Some of them require heating and co-solvent to dissolve in CO₂. -Their CO₂ viscosity-enhancing ability decreases at elevated temperatures. -Small molecule CO₂-rich gels are more viscous and difficult to flow. -Fluorinated small molecules are costly and environmentally unfriendly.
Surfactants	Micelles formation and self-assembly into network structures with stable foam.	<ul style="list-style-type: none"> -Reduce the CO₂ relative permeability. -Excellent foaming agents for CO₂ in water-sensitive formations. -Reduce the capillary forces and IFT in the reservoir formation. 	<ul style="list-style-type: none"> -Surfactants' composition is thermodynamically unstable. -Surfactants have strong adsorption affinity on porous rock formations. -Large quantity is needed to compensate for the loss due to adsorption.
Nanoparticles	Dispersed in CO ₂ -aqueous solution and stabilize the CO ₂ foam.	<ul style="list-style-type: none"> -Alter the wettability and reduce the IFT. -Maintain homogeneity of the CO₂ solutions. -Prevents precipitation in the bulk fluids at normal reservoir conditions. 	<ul style="list-style-type: none"> -A substantial amount is needed to achieve a significant CO₂ viscosity increase. -Some nanoparticles might agglomerate under harsh reservoir conditions. -Fluorinated-based nanoparticles are costly and environmentally unfriendly.

Table 7
Polymers and surfactants pilot field tests for CO₂ mobility and conformance control.

CO ₂ thickener	Field name	Application	Research findings	Reference
Chromic-acetate acrylamide polymer gel	Rangely Weber	CO ₂ mobility control.	CO ₂ production was reduced and the oil output was increased.	[241]
Cross-linked polyacrylamide gel	Bati Raman	CO ₂ flood conformance improvement.	Increased CO ₂ sweep efficiency and oil production.	[242]
Partially hydrolyzed polyacrylamide (PHPA) gel	SACROC	CO ₂ flood conformance improvement.	Reduced CO ₂ production and increased oil output.	[243]
Surfactant (Alipal CD-128)	Rock Creek	CO ₂ mobility control.	Reduced CO ₂ injectivity problems, but no oil displacements.	[244]
Surfactant (Chaser CD-1040)	Rangely Weber	CO ₂ mobility control.	CO ₂ mobility was reduced and oil production was increased.	[245]
Surfactant (Chaser CD-1040)	North Ward-Estes	CO ₂ flood conformance improvement.	CO ₂ injectivity problems were reduced and the oil recovery was increased.	[246]
Surfactant (Chaser CD-1045)	EVG-San Andres Unit	CO ₂ mobility control.	Delayed CO ₂ breakthrough and increased oil output.	[247]
Alipal CD-128/ Chaser CD-1045	East Mallet Unit	CO ₂ mobility control.	CO ₂ production was decreased and the oil production rate was increased.	[248]
Alipal CD-128/ Chaser CD-1045	McElmo Creek Unit	CO ₂ mobility control.	CO ₂ production was decreased and the oil production rate was increased.	[248]
ELEVATE™ Foam	SACROC	CO ₂ flood conformance improvement.	CO ₂ mobility was reduced and oil production was increased.	[249]
Huntsman L24-22/C12-14 E022	East Seminole	CO ₂ mobility control.	CO ₂ mobility was reduced after surfactant treatments.	[250]

of 90,000 bbls and a reduction in the gas-to-oil ratio (GOR) from 1000 to 100 for 1.5 years.

Surfactants have been successfully utilized in several pilot field tests for CO₂ mobility and conformance control. Heller et al. [244] were the first research group to report the CO₂ foam pilot field test which was implemented at the Rock Creek field in Roane County, West Virginia, USA. The results from their pilot test demonstrated that the CO₂ foam produced using Alipal CD-128 surfactant was effective in reducing CO₂ injectivity problems. Jonas et al. [245] conducted a CO₂ foam pilot test using CO₂-soluble surfactant in the Rangely Weber Sand field, North-western Colorado, USA. The results obtained showed that the CO₂ mobility was reduced and the oil production was increased after the foam treatment, and the foam remained stable in the reservoir throughout the evaluation period. Chou and colleagues [246] have reported on the use of foam generated from surfactants to improve CO₂ injectivity in the North Ward-Estes field, Winkler County, Texas, USA. The results from the pilot test demonstrated that the CO₂ injectivity problems were effectively reduced by 40 to 85% and the oil production rate was increased in the offset producers. Martin et al. [247] conducted a CO₂ foam field test to control CO₂ mobility in the East Vacuum Grayberg/San Andres Unit, Lea County, USA. The results obtained from the field test showed that the CO₂ mobility could be reduced by 1/3 using foam compared to CO₂ injection alone. Hoefner and Evans [248] have reported four developmental wells CO₂-foam field trials in carbonate reservoirs (well 31 and 68 in East Mallet Unit of Hockley County-Texas, and well P-19 and R-21 in McElmo Creek Unit of San Juan County-Utah), USA. The results from the field treatments demonstrated a substantial reduction in CO₂ mobility in two of the four field trials (well 31 and P-19) and an increased overall oil production rate by 10% to 30%.

Furthermore, Sanders et al. [249] conducted a CO₂-foam pilot test to control CO₂ mobility at Scurry Area Canyon Reef Operational Committee (SACROC) field in west Texas, USA. The results obtained from the pilot test showed a significant reduction in CO₂ mobility by over 50% compared to CO₂ injection alone and a substantial increase in oil production (30%) was achieved at offset oil production wells. Mukherjee et al. [252,253] have reported a CO₂ foam pilot test to address the CO₂ mobility issues in the Salt Creek field, Natrona County, USA. The pilot test results depicted a significant reduction (40%) in CO₂ mobility and were used to develop a model that anticipated an increase in the oil production rate. Alcorn research group have implemented a CO₂ foam pilot test using non-ionic surfactant (linear ethoxylated alcohol) in East Seminole Field,

Permian Basin, west Texas, USA [250]. The results obtained from the pilot test showed a CO₂ mobility reduction of 70% using surfactant compared to CO₂ injection alone. Mirzaei et al. [30] have conducted a CO₂ foam pilot test using a CO₂-soluble surfactant namely ELEVATE™ to address the CO₂ conformance problems in hydrocarbon reservoirs in West Texas, USA. The pilot test results revealed that the utilized foam had successfully reduced the conformance problems in the studied area. However, the oil production rate response was insufficient because the pilot test was performed without a fully developed simulation model. Moreover, the research findings from polymer and surfactant pilot field tests are promising; nevertheless, further research work needs to be conducted to find effective and affordable CO₂ thickeners for EOR field applications.

Screening criteria for designing CO₂ thickeners

The screening criteria for designing CO₂ thickeners principally depend on the reservoir properties, working conditions, the solubility of thickener in CO₂, the amount of co-solvent required, environmental concerns, and availability and cost of thickener [37,254]. The solubility of thickeners in CO₂ is the prime factor to consider when designing CO₂ thickeners. Polymer solubility in CO₂ is affected by the strength of covalent bonds, the degree of crystallinity or amorphous of the polymer, and the intermolecular forces between the polymer chains [57]. Previous studies have reported that, polymers with low surface tension, low cohesive energy density, low solubility parameter, low polarizability, low glass transition temperature (T_g), and high free volume exhibit high solubility in CO₂ [57,58,255]. A good CO₂ thickener must be soluble enough in CO₂ at reservoir conditions without the need for co-solvents and must increase the viscosity of CO₂ by a factor of 10–100 folds in a concentration of less than 1wt% [154]. The thickener must be less expensive, non-toxic, highly CO₂ soluble without heating the mixture, and environmentally friendly. The thickener must be soluble enough in dense CO₂ but insoluble in both brine solution and crude oil at reservoir conditions [154]. The thickener must be tested in CO₂ instead of organic liquids such as light alkanes during an initial screening stage. The thickener must be designed/synthesized specifically for application in CO₂ systems by incorporating important CO₂-philic/phobic functionalities instead of evaluating hundreds of commercially available chemical compounds. However, these criteria are not the rule of thumb; they just serve as a baseline for making decisions during

the initial screening stage. For example, one may design a CO₂ thickening agent that is relatively inexpensive but requires a large volume of a co-solvent to dissolve and thicken CO₂, hence, making the process not practical for CO₂-EOR field application.

Challenges facing CO₂ thickening technologies

The major challenge towards achieving effective CO₂ thickeners is the low solubility of thickeners in dense CO₂ resulting in a large volume of co-solvent requirement or expensive CO₂-philic groups [256,257]. Many conventional thickeners particularly polymers are less soluble in CO₂ unless higher pressure that exceeds the minimum miscibility pressure (MMP) and co-solvent are used [27,41]. High molecular weight polymers are reported to have low solubility in CO₂ whereas low molecular weight polymers are reported to have high solubility in CO₂ with minor viscosification [75,258]. The majority of the co-solvents used to thicken CO₂ are environmentally unfriendly and highly expensive; thus, making the process not practical for CO₂-EOR field applications [259]. Fluorinated-based CO₂ thickeners are environmentally unfriendly, very expensive and have higher adsorption affinity on porous rocks hence reducing their CO₂ thickening ability [1]. Small molecule CO₂-rich gels are more viscous and difficult to flow through the porous rocks at normal reservoir conditions but also require heating to maintain solution homogeneity. Nanoparticles have shown promising performance in stabilizing CO₂ foam; however, they tend to precipitate/agglomerate under harsh reservoir conditions. Despite many decades of intensive research work on CO₂ thickeners, an effective and affordable CO₂ thickener that is free from these challenges has not yet been found. Therefore, more research studies need to be conducted to overcome these challenges and to find appropriate CO₂ thickeners for EOR field applications.

Future research directions on CO₂ thickeners

Despite many years of intensive research work on CO₂ thickeners, an effective, affordable and environmentally friendly CO₂ thickener that can dissolve in CO₂ at lower concentrations and increase the CO₂ viscosity significantly to a level similar to that of crude oil without the addition of co-solvents has not yet been found. Future research studies on CO₂ thickeners must focus on designing and synthesizing less expensive, non-fluorous, non-toxic, environmentally friendly and highly soluble CO₂ thickeners that require less or no volume of co-solvent to achieve higher CO₂ viscosity [53,260,261]. Recently, researchers have drawn more attention to biopolymers and their derivatives as greener chemical agents for EOR operations [61,262]. Biopolymers such as starch, sugar acetates, xanthan gum and guar gum which are abundant and naturally available are of great interest for future research in CO₂ thickeners [61,263]. Polymeric compounds containing acetate, ether, carbonate, and carbonyl functionalities have currently been reported as attractive CO₂-thickening candidates for future research studies [107,109,260,264]. Currently, poly(vinyl acetate) is the only high molecular weight, non-fluorous and most CO₂ soluble polymer that demonstrated a significant CO₂ thickening ability over a broad spectrum of temperatures [129]. Future research studies should also consider incorporating more than one CO₂ thickening agent, specifically, nanoparticles can be incorporated with polymers or surfactants to increase the CO₂ viscosity and enhance oil recovery. Furthermore, the molecular structure of the CO₂ thickener should be critically designed to achieve a structural balance between CO₂-philic/CO₂-phobic groups to enhance the solubility of the thickener and increase the viscosity of CO₂ [265].

Conclusion

Several chemical compounds such as polymers, surfactants, small molecules and nanoparticles have been designed and investigated as CO₂ thickeners. Currently, none of these chemicals can be regarded as effective and affordable CO₂ thickeners for EOR field applications. The results obtained from laboratory investigations, simulation studies and molecular dynamics modelling revealed that PDMS, polyFAST, PFOA, PVAc and P-1-D remain the most technically viable polymers for CO₂ mobility control. However, their field applications are not practical because of their high costs, large amount is needed to produce satisfactory results and environmental constraints. Small-molecule compounds both fluorinated, semi-fluorinated and non-fluorinated have been identified in previous literature as CO₂ thickeners. They are capable of viscosifying the CO₂ by up to 300-fold at ambient temperatures (298–315 K). The experimental studies on microemulsions prepared using CO₂ and surfactants demonstrated a substantial increase in CO₂ viscosity implying a reduction in CO₂ mobility. The application of nanoparticles for CO₂ mobility control has provided an alternative method for generating stable CO₂ foam which contributes to an increase in CO₂ viscosity. Despite many years of intensive research work on CO₂ thickeners, most of the attempts are either laboratory or simulation studies based with a limited number of pilot field tests. Moreover, further research work with an emphasis on pilot field tests needs to be conducted to identify effective and affordable CO₂ thickeners for EOR field applications.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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