

**REVIEW**

A Research Progress of CO₂-Responsive Plugging Channeling Gels

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ABSTRACT

In the heterogeneous reservoirs, CO₂ flooding easily leads to CO₂ gas channeling, which can seriously affect sweeping efficiency and reduce oil recovery. After thoroughly investigating the advantages and shortcomings of various CO₂ plugging technologies, this paper focuses on the feasibility of improving conventional water-alternating gas (WAG) through CO₂-responsive gel materials. Based on the different chemical reaction mechanisms between the unique chemical structure and CO₂, changes in the material's physical and chemical properties can respond to CO₂. The feasibility of utilizing these property changes for CO₂-responsive plugging is explored. Various CO₂-responsive gels and gel nanoparticles have been extensively researched in different fields, such as energy, medicine, and biology. This paper surveys the molecular structures, chemical compositions, response mechanisms, and changes of these CO₂-responsive gels, aiming to draw insights into the carbon dioxide-enhanced oil recovery (CO₂-EOR) field. Finally, the key issues and future development direction of CO₂-responsive plugging gels were analyzed.

KEYWORDSGas channeling; sweep efficiency; CO₂-responsive gel; CO₂-EOR

1 Introduction

The carbon capture, utilization, and storage (CCUS) are the most practical and feasible carbon reduction technologies. CO₂ possesses unique physicochemical properties, is widely available, easily accessible, and safe. Currently, it is extensively used for EOR, significantly improving the oil recovery of heavy oil reservoirs and low-permeability oil fields. Therefore, CCUS-EOR technologies, represented by CO₂ flooding, have broad prospects for both carbon reduction and resource utilization [1]. Research indicates that due to CO₂'s low minimum miscibility pressure (MMP), it is easily miscible with crude oil, causing it to expand, reducing capillary and flow resistance. Consequently, this significantly enhances the flow capacity of crude oil [2].

Gas channeling is a significant issue in the CO₂ flooding process, leading to a decrease in CO₂ sweep efficiency and a noticeable reduction in oil recovery. See Fig. 1a, the main cause of gas channeling is the severe heterogeneity in the reservoir. Injected CO₂ is prone to channeling along the high seepage zone such as fractures and large-pore channels, greatly diminishing the efficiency of CO₂ sweep. Because crude oil has a much higher viscosity than CO₂, its mobility differs significantly, making it susceptible to viscous fingering, which hinders efficient oil displacement [3].



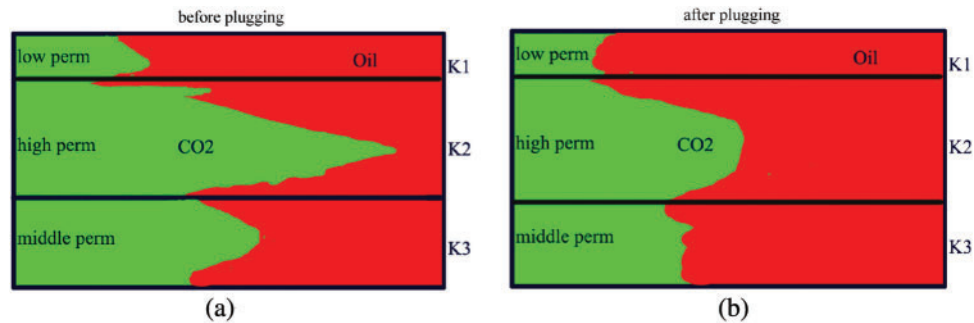


Figure 1: CO₂ gas channeling

Currently, there are two main methods to inhibit CO₂ channeling and enhance displacement phase sweeping efficiency. See Fig. 1b, the first method involves plugging highly permeable channels to suppress the advancement of CO₂ along preferential pathways, utilizing techniques such as gel plugging and foam plugging. The second method involves increasing the viscosity of the displacing phase to reduce the mobility ratio, employing techniques like WAG and CO₂ thickening.

1.1 Current Research Status of CO₂ Plugging Channeling Technologies

1.1.1 WAG Technology

WAG technology is a process that uses alternating slugs by injecting gas and water, which enhances gas sweep efficiency and minimizes the required gas volume [4]. The CO₂-WAG injection process is seen in Fig. 2 [5]. WAG combines the advantages of water and gas injection, where water improves macroscopic sweep efficiency, and gas, due to its high permeability, enhances microscopic sweep efficiency. Additionally, CO₂ has a relatively low minimum miscibility pressure (MMP) and is easily miscible with light crude oil. Injecting water increases reservoir pressure above the MMP of CO₂, making it more soluble in crude oil and reducing the relative permeability of CO₂ [6–8]. As early as the 20th century, CO₂-WAG injection technology has been successfully applied in some sandstone reservoirs in the United States. References [9–11] reported the performance of the CO₂-WAG project in the U.S. Gulf coast reservoir. These projects have achieved success, the oil production has significantly improved. However, CO₂-WAG technology also has some drawbacks. Firstly, due to the higher density and viscosity of water compared to CO₂, water tends to preferentially fill the high permeability layers in the reservoir, forcing CO₂ to change its flow direction towards the low permeability layers, known as gravity segregation. Secondly, the presence of CO₂ makes the reservoir acidic, leading to corrosion of pipelines during the injection process and precipitation of polar substances such as asphaltene that are insoluble in crude oil, causing blockages in flow channels [12]. Thirdly, water entering the formation increases the water saturation near the wellbore, increasing capillary resistance at the oil-water interface in the pores, a shielding effect that undermines the efficiency of microscopic displacement [13,14]. The presence of the Jamin effect can lead to water blocking [15], which is unfavorable for oil recovery. Lastly, WAG process design and optimization parameters are complex, with gas-water ratio, injection pattern, injection rate, injection volume, and slug size all impacting the operational effectiveness [16].

1.1.2 CO₂ Thickening Technology

CO₂ thickening technology utilizes polymers to increase the viscosity of CO₂, thereby reducing its mobility and weakening the effect of viscous fingering. As the density of CO₂ is much lower than that

of formation water and crude oil, using CO₂ as a displacing phase can easily lead to phenomena such as gravity segregation and viscous fingering, resulting in a significant amount of residual oil. see Fig. 3 below [17]. Currently, there are two main methods for directly thickening CO₂. One approach involves using organic solvents to increase the solubility of conventional thickening agent compounds in the CO₂ phase. The feasibility of this method depends on the ability of the polymer/solvent combination to increase the viscosity of CO₂. Another method involves using additives with both hydrophilic and hydrophobic properties. Polymer segments can form supramolecular solution structures under binding conditions [18]. Polymers currently used in CO₂ thickening technology mainly include fluorinated polymers, silicone polymers, alkanes polymers, etc. [19,20]. Silicone polymers, fluoropolymers and alkanes polymers have achieved the thickening effect [21–24]. However, low molecular weight alkanes polymers, including poly (vinyl ethyl ether) (PVVE) and poly-1-decene (PID), it has a little CO₂ thickening [25,26].

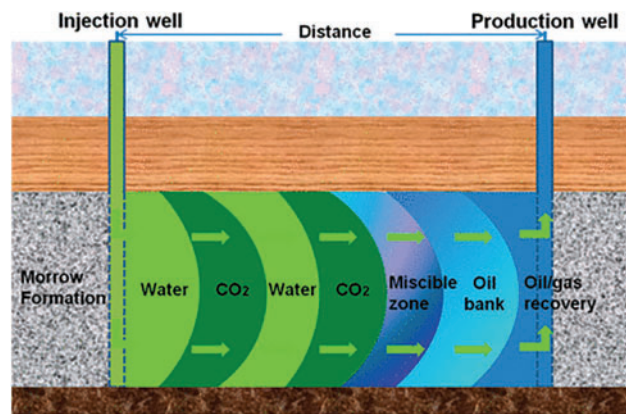


Figure 2: CO₂-WAG injection process under mixed-phase pressure (modified from Dai et al. [5])

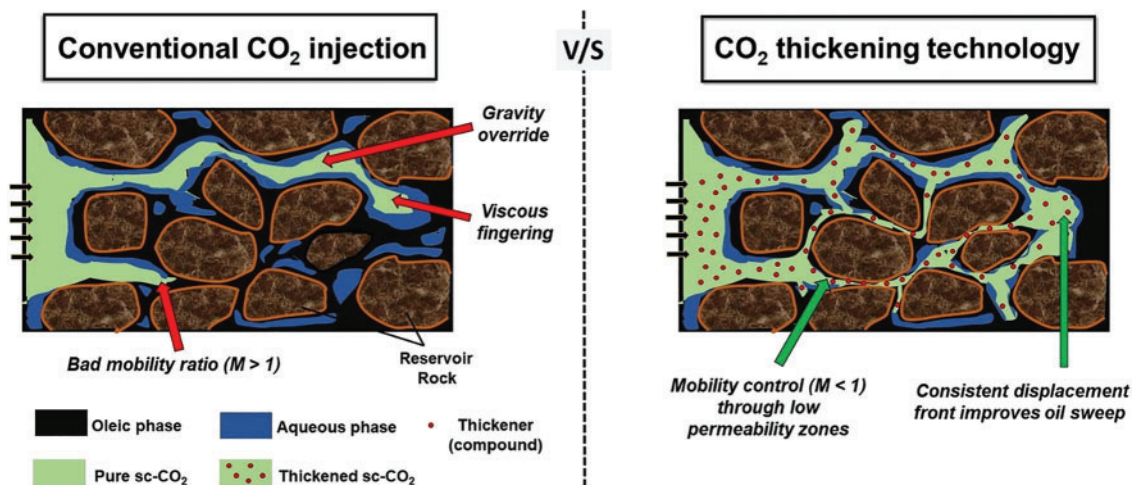


Figure 3: Illustration of challenges during CO₂ injection such as viscous fingering and gravity override (modified from Pal et al. [17])

However, direct CO₂ thickening technology also faces significant challenges. Firstly, although fluorinated and silicone polymers can achieve a certain thickening effect, silicone polymers have poor

solubility and generally require a large amount of co-solvent, need higher temperature and pressure, making them challenging for widespread use. Secondly, fluorinated polymers have issues of high toxicity and poor degradability, which can have adverse environmental effects and are not conducive to practical production. Thirdly, alkanes polymers, despite having good CO₂ solubility, face the problem of decreasing solubility with increasing carbon chain length, resulting in high costs [19]. Therefore, developing environmentally friendly, cost-effective, non-toxic, and highly effective thickening agents remains a significant challenge.

1.1.3 Gel Plugging Technology

Gels in formation water can increase viscosity or swell, thereby plugging high-permeability layers and altering the flow direction of gases, enhancing the sweep efficiency of the displacing phase. It is mainly divided into two types: one is *in-situ* gel formation, composed of monomers or polymers, cross-linking agents, and other additives in a solution. When injected into the formation, it forms a gel under reservoir conditions, plugging the high-permeability layers. The other type is ground pre-crosslinked gels which mainly include pre-crosslinked particle gels (PPGs), polymer microspheres, and colloidal dispersion gels (CDGs) [27]. Under reservoir conditions, these particles gradually swell and plug the high-permeability Bai et al. [28]. Lantz et al. [29] reported the use of partially hydrolyzed polyacrylamide (HPAM) and Cr³⁺ cross-linking agent to treat some oil reservoirs, results showed the oil recovery increased. Zhou et al. [30] used free radical polymerization to prepare an acid-resistant pre-formed particle gel (AR-PPG). Results showed that AR-PPG had a swelling ratio of 1.41 in acidic environments. Compared to traditional PPG, AR-PPG exhibited better swelling performance, shear resistance, and viscoelasticity in acidic environments, achieving a plugging rate of 70.8% in CO₂-driven low-permeability micro fractured cores.

Traditional gel plugging technology is mainly used for water plugging and has poor adaptability to CO₂ channeling. For example, conventional *in-situ* gels have poor acid resistance, fast cross-linking under acidic conditions, high dehydration rates, and commonly used cross-linking agents are environmentally toxic [2]. Regarding traditional preformed particles, the matching relationship between particle size and formation pore throats directly affects the injectivity of the plugging agents. Acidic and high-temperature, high-salinity environments can accelerate particle degradation and dehydration [31]. The degradation products may contain ketone, aldehyde, carboxylic ester groups, and monomer units, which can adsorb onto mineral surfaces, causing harm to the reservoir [32] and they are poorly adapted to low-permeability reservoirs [16]. Although polymers with temperature, salt, and acid resistance have been developed, their high costs make them economically unfeasible for widespread use.

1.1.4 Foam Plugging Technology

Foam, with greater effective viscosity than gas, can alleviate gravity override and viscous fingering during gas injection, and by increasing flow resistance, it prompts the injected fluid to divert from high-permeability layers to low-permeability layers [33]. The improvement of the long-term stability of CO₂ foams has been the subject of extensive research efforts. Such approaches include the use of surfactants (cationic, anionic, non-ionic, and zwitterionic), viscoelastic surfactants (VES), polymers, and nanoparticles (NPs) [34]. There are generally three methods of foam injection: generating foam externally and injecting it into porous media, injecting surfactant and CO₂ simultaneously into porous media to form foam, and alternating injection of surfactant and CO₂ (SAG).

Using surfacants to control the mobility of CO₂ Studies showed that foam could enhance gas displacement by reducing permeability, altering mobility ratio, and preventing channeling [35–37]. The reference [38–40] have investigated the utilization of nanoparticles for enhancing foam stability, the stabilization time is evidently extended. Additionally, foam plugging technologies have also been successfully applied in some North American oilfields. Some CO₂ foam tests that all decreased CO₂ injection capacity, effectively altering the mobility of CO₂ [41,42]. However, foam plugging technology also faces the following challenges:

1. Poor stability and oil resistance of foam systems, leading to a short effective period;
2. Poor injectivity due to additional resistance during foam injection caused by gas Jamin effect, increasing pump pressure and construction difficulty;
3. High costs due to the decreased actual injection volume caused by surfactant adsorption during the oil displacement process, requiring continuous addition of surfactant to ensure overall construction effectiveness, thus increasing economic costs;
4. Cationic surfactants, while exhibiting good foaming properties in the acidic environment induced by CO₂ flooding, are mainly nitrogen-containing amine derivatives, posing toxicity risks to humans and the environment. Currently, many nanoparticles have been developed to enhance the stability of foam, which can delay bubble coalescence and improve foam stability. Reference [43] developed a CO₂ foam system suitable for high-temperature and high-salinity conditions by adding 1% nano-silica to the system. The results indicate that the half-life of the foam was extended by 14 min. However, there is a problem with nanoparticle aggregation, which is not conducive to achieving the maximum performance of nanoparticles [16]. Therefore, there are certain challenges and difficulties in developing highly effective surfactants for CO₂ flooding.

This article first investigates the laboratory research and field applications of various CO₂-plugging technologies. After analyzing the advantages and disadvantages of existing plugging technologies, the article proposes technical measures using CO₂-responsive gel materials for selectively plugging CO₂. Subsequently, Based on the different chemical reaction mechanisms between the unique chemical structure and CO₂, the article explores the mechanisms behind CO₂-responsive plugging. Finally, the research surveys the current applications of various CO₂-responsive gels and analyzes their potential applications in the field of improving crude oil recovery by preventing CO₂ channeling.

CO₂-responsive gel materials are environmentally sensitive intelligent plugging materials. Applying them in the development of oil-gas fields holds the potential to address the shortcomings of current CO₂ plugging technologies, making a significant contribution to CO₂ sequestration and resource utilization.

2 CO₂-Responsive Mechanism

2.1 Reaction Mechanism of CO₂ with Specific Functional Groups

The CO₂ response mechanism mainly refers to the reversible changes in the properties of compounds after the introduction and removal of carbon dioxide in the system. This is achieved by involving only the introduction and release of inert gases such as CO₂ and N₂ in the process, thereby avoiding the generation of by-products and system contamination. Compounds with this kind of stimuli-responsive behavior controlled by a “green” switch have broad applications in sensors, surface adhesives, and drug delivery carriers. These compounds contain specific functional groups that can react with CO₂ [44], primarily including amine groups, carboxyl groups, imine groups, guanidine

groups, etc. The reaction mechanisms can generally be classified into the following four types, see Figs. 4–8 below.

(a) Tertiary amine reacts with CO₂ and water to form bicarbonate, see Fig. 4

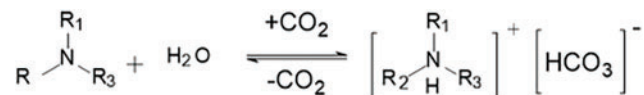


Figure 4: Reaction mechanism of tertiary amine group with CO₂

Due to the basicity and water solubility of tertiary amines, upon the introduction of CO₂ under acidic conditions, the amine groups of tertiary amines will be protonated by a large number of protons produced from the ionization of carbonic acid. As the ionization degree of tertiary amines increases, their hydrophilic ability strengthens. When inert gases such as N₂, Ar, etc., are introduced into the system to remove CO₂, this reaction will reverse, leading to a decrease in the hydrophilic properties of tertiary amines. Therefore, cationic hydrogels containing tertiary amine groups exhibit a lower degree of swelling when not in contact with CO₂, allowing them to smoothly enter high-permeability layers. Upon contact with infiltrating CO₂, they moderately swell to achieve plugging.

(b) Carboxylic ion reacts with CO₂ and water to form carboxylic acid, see Fig. 5

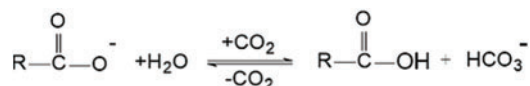


Figure 5: Reaction mechanism of carboxylic ion with CO₂

Carboxylic ions exhibit low dissociation under acidic conditions when CO₂ is introduced, rendering them electrically neutral. The O-H bond in the carboxyl group, however, possesses strong polarity and can form hydrogen bonds with other carboxylic acid groups. These dual effects collectively induce volume contraction in gels containing carboxylic acid groups. When the system's pH is higher than the pK_a of carbonic acid (6.35), the proton dissociation capacity of carboxylic acid groups significantly increases, transforming into carboxylate ions. This enhances the hydrophilicity of the gel, causing the gel network to swell, and resulting in a noticeable increase in volume. Therefore, anionic hydrogels containing carboxylic acid groups are only suitable for plugging under alkaline conditions and are not suitable for plugging CO₂.

(c) Amidine/Guanidine reacts with CO₂ and water to form the corresponding amidine or guanidine salt, see Figs. 6, 7

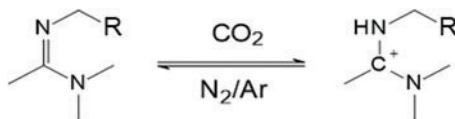


Figure 6: Reaction mechanism of amidine with CO₂

The secondary amino group in the imine or guanidine contains lone pair electrons, which, upon reacting with CO₂ and water, form a Lewis acid-base pair with the generated carbonic acid. This triggers an electrophilic reaction, producing the corresponding imine salt or guanidine salt. The

significantly increased hydrophilicity induces water absorption and swelling in gels containing imine or guanidine groups, achieving plugging. When the gel is not in contact with CO₂, deprotonation occurs, leading to volume contraction, which helps improve the injectivity of the gel.

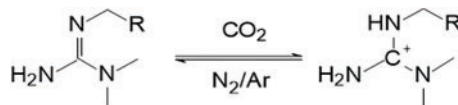


Figure 7: Reaction mechanism of guanidine with CO₂

(d) Primary amines react with CO₂ to form carbamates, see Fig. 8

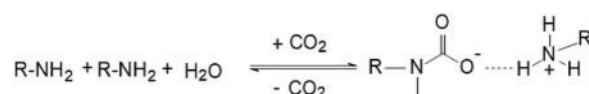


Figure 8: Reaction mechanism of primary amine with CO₂

At room temperature and pressure, CO₂ can bind with two molecules of primary amines, forming the anionic aminomethanoate and protonated cationic primary amine. These two entities form a salt through electrostatic interactions. When the system is heated, the aminomethanoate salt decomposes, releasing CO₂ gas from the system, and allowing the system to reversibly return to its initial state. Therefore, primary amines can block CO₂ infiltration channels by generating precipitates.

2.2 Gel Plugging Channeling Mechanism

CO₂-responsive plugging shares similarities with non-responsive plugging. Both involve physico-chemical changes, such as expansion, thickening, adsorption, solidification, etc., occurring in underground high-permeability layers to block advantageous pathways for gas channeling. However, there are clear differences. The conventional non-responsive gel change process is typically triggered by reservoir temperature, primarily plugging water or gas fluid channeling pathways. In contrast, the responsive gel change process is stimulated by gas channeling of CO₂, selectively plugging CO₂ infiltration pathways. Therefore, gels with CO₂ responsiveness are more suitable for selectively plugging CO₂ gas channeling under injection conditions.

2.2.1 Swelling Mechanism

Gel particle water absorption and swelling refer to the diffusion of water molecules into the three-dimensional network inside gel particles upon contact with water. This causes the original polymer chains to transition from coiled to extended, increasing the system's water content and significantly expanding its volume.

The swelling performance of gel particles (including swelling rate and swelling ratio) depends mainly on the hydrophilic properties of polymer segments, gel size, reservoir temperature, mineralization, and other factors. The slow expansion of gel particles effectively resolves the contradiction between injectivity and plugging performance. Currently used particle-sized gels and polymer microspheres operate on this plugging mechanism [45–48].

2.2.2 Increasing Viscosity Mechanism

Generally, water-soluble polymer viscosity is low, resulting in weak plugging capabilities. By adding a crosslinking agent to low-concentration polymer solutions that delays the crosslinking reaction (such as chromium ions, phenol-formaldehyde resins, etc.), or adding initiators to monomer solutions, a gel with higher viscoelasticity can slowly undergo sol-gel transition underground at high temperatures. This forms a gel with higher viscosity and plugs the high-permeability layer, forcing injected water and gas into low-permeability layer pores, thereby improving the displacement phase's sweep efficiency. Similar to gel particles, this delayed thickening characteristic effectively addresses the contradiction between the injectivity and plugging performance of plugging agents. Currently used chromium gels, phenol-formaldehyde resin gels, *in-situ* gels, etc., operate on this plugging mechanism [49–51].

2.2.3 Adsorption Mechanism

Some gel materials or surfactants contain a large number of polar groups, allowing for the formation of physical or chemical adsorption on the pore surfaces of high-permeability layers through electrostatic interactions, chemical reactions, etc. This reduces pore size, increases flow resistance of the displacing phase, or alters the wettability of the rock, thereby promoting injected water and gas into low-permeability layers, expanding sweep efficiency. However, this adsorption action inevitably causes short-term or permanent damage to the reservoir. Currently used cationic polyacrylamides, cationic surfactants, etc., operate on this mechanism [52].

In summary, plugging materials, through physicochemical changes such as expansion, thickening, adsorption, etc., in high-permeability layers experiencing fluid flow, form plugging zones with certain pressure-holding capabilities. This is the primary means of inhibiting the infiltration of the displacing phase and expanding sweep efficiency. However, these plugging materials generally lack selectivity for CO₂. Some plugging materials, such as polyacrylamide gels, may exhibit unfavorable changes in performance, such as viscosity reduction and volume contraction, under acidic conditions. Therefore, traditional plugging materials may not necessarily be suitable for selectively plugging CO₂ infiltration.

3 CO₂-Responsive Gel

Gel materials, due to their high viscosity or mechanical strength and strong deformability, are commonly used plugging agents in water and gas control in oil and gas extraction. To achieve selective CO₂ plugging by gels, CO₂-responsive functional groups are introduced into polymer molecules. Based on the reaction mechanism between CO₂-responsive functional groups and CO₂, various CO₂-responsive gel materials have been developed in different application areas. In this article, we investigate several types of CO₂-responsive gels based on cross-linking mechanisms, including chemical crosslink and physical crosslink.

3.1 Chemical Crosslink Gels

3.1.1 Double-Bond Crosslink Gel

Double-bond crosslink agents are generally added during polymer preparation and participate in monomer copolymerization. The most common double-bond crosslink agents generally can be divided into two categories: N, N-methylene bisacrylamide (MBA), and N, N'-bis(acryloyl)cystamine (BACy). MBA is known for its advantages of structural stability, easy preparation, and convenient cross-linking degree adjustment [53]. Therefore, it is widely used in oil and gas development, preparation

of photosensitive materials, and separation of biopolymer compounds (proteins, peptides, nucleic acids), etc. [54–57]. However, it is not convenient for underground use and is generally employed in the preparation of pre-crosslinked gel particles. The other double-bond crosslink agent is BACy, It is often used as a crosslinking agent to adsorb dyes and sewage materials with strong stability, incompatible with strong oxidants, and insoluble in water [58,59].

This article investigates CO₂-responsive gel particles of different sizes. Gel particles are colloidal gel particles composed of chemically cross-linked three-dimensional polymer networks. Based on their different sizes, they can be classified into nano-gels, micro-gels, and large particle gels. Nanogel (particles with sizes <100 nm), microgel (particle size between 100 nm and 100 μm), and large particle gel (particles with sizes >100 μm) [60]. These are suitable for plugging different-sized fluid flow channels.

Gel particles can swell under certain conditions, and their swelling performance is influenced by cross-linking density, elasticity, and the nature of the polymer chain segments forming the gel. Compared to large gels, gel particles have significant advantages. Firstly, they have a larger surface area, resulting in a faster response compared to large gels. Secondly, the viscosity of micro-gel suspensions depends mainly on the viscosity of the dispersed phase and the concentration of micro-gels, making their viscosity much lower than that of soft solid-like large gels. Similar to conventional micro-gels, responsive micro-gels are typically synthesized through precipitation polymerization and inverse emulsion polymerization and have been widely used in areas such as dye adsorption, biomedical applications, and enhanced oil recovery [60]. The applications of double-bond crosslink gels are seen in Table 1.

Table 1: Application of double-bond crosslink gels

Author, year	Crosslink agent	Responsive functional group	Main composition	Responsive performance of gel (when injecting CO ₂)	Reference
Pu et al., 2021	MBA	Tertiary amine	AM, DMAEMA	Injection pressure increased from 5.0 kPa to 0.342 MPa, the plugging efficiency of 99%, and the EOR increased by 23.1% Original Oil in Place (OOIP).	[53]
Sun et al., 2018	MBA	Secondary amine	AM, DEAEMA	The SR reaches 27.5~28.	[54]
Zhang et al., 2018	MBA	Tertiary amine	AM, DMAEMA, acrylic acid (AA)	When the proportion of DMAEMA in the response monomer ratio ranged from 4.1%~23.3%, it would clearly swell.	[55]

(Continued)

Table 1 (continued)

Author, year	Crosslink agent	Responsive functional group	Main composition	Responsive performance of gel (when injecting CO ₂)	Reference
Zhang et al., 2022	MBA	Heterocyclic azoles	Tetrabutylphosphine 4-styrene sulfonate+ 1-vinyl-1H imidazole	All follow LCST-type volume phase transitions in water, dimethylacetamide, and water/dimethylacetamide mixtures.	[56]
Chen et al., 2018	MBA	Carboxyl	AM, AA	The particle size was positively correlated with the pH of the system.	[57]
Corona Rivera et al., 2022	BACy	Tertiary amine	AM, N, N'-dimethylaminoethyl methacrylate (DAEMA)	Adsorption capacity of 1413 mg/g for the Alizarin Red.	[58]
He et al., 2021	BACy	Tertiary amine	AM, 2-(Diethylamino) ethyl methacrylate (DEA)	Adsorption capacity was up to 1537 mg/g for metanil yellow (MY).	[59]

3.1.2 The Applications of Other Types CO₂-Responsive Microgels

Based on the reaction mechanism of different functional groups to achieve reversible volume expansion and contraction through alternating treatment with CO₂ and N₂ and play the properties of the materials [61–65].

Rabiee et al. [61] developed cationic microgels of 2-(diethylamino) ethyl methacrylate (DEAEMA) and 2-(dimethylamino) ethyl methacrylate microgels (DMAEMA). As the CO₂ will induce the tertiary amine matrix derivatization, enhance the hydrophilicity, and lead to the expansion of microgel due to the effect of electrostatic repulsion. Hence, the microgel could absorb water and dehydrate through CO₂/N₂ bubbling and Water recovery by 50% can be achieved for these microgels. But N₂ bubbling will deprotonate until the microgel is uncharged and appears as volume shrinkage. Furthermore, CO₂/N₂ bubbling will change the surface charge of microgels, which will affect the swelling/shrinking properties and water absorption/dehydration capacity of microgels.

Xu et al. [62] synthesized CO₂-responsive waterborne polyurethane-polydimethylaminoethyl methacrylate (WPU-co-PDM) emulsion by free radical copolymerization. The particle size of the emulsion increased from 80 nm at 25°C to 180 nm at 50°C, after CO₂ bubbling into the emulsion, Upon bubbling with N₂, the particle size returned to the original size, and it showed CO₂ responsiveness. As the temperature increased, the ionized quaternary ammonium salt underwent “deprotonation,” transforming hydrophilic quaternary ammonium cations into hydrophobic tertiary amine structures. Therefore, emulsification could be achieved by bubbling CO₂ or cooling, while de-emulsification could be achieved by bubbling N₂ and heating.

Shieh et al. [63] used multi-responsive amphiphilic block copolymers containing methacrylic acid (MAA) and the emulsifiers 2-dimethylaminoethyl methacrylate (DMAEMA), two-way CO₂ stimuli-responsive PMMA latex particles were prepared via aqueous emulsion polymerization. By

tuning the length of the PDMAEMA block in the copolymer the PMMA-surfaced latex particles can exhibit a two-way CO₂ response between dispersion and precipitation at acidic (pH = 2) and alkaline (pH = 12).

In 2023, Al Akoumy et al. [64] synthesized core-shell nanoparticles as gas-controlled reduction catalysts for 4-nitrophenol (4-NP) and nitrobenzene (NB). PDEAEMA, a CO₂-responsive crosslinked polymer, adjusted the catalytic reaction rate through the reversible switching of CO₂/N₂ bubbling. For the reduction of 4-NP, it is possible to turn on and turn off the reaction through alternating bubbling of the two gases. CO₂ can promote a faster reaction of 4-nitrophenol (4-NP), while N₂ facilitates the faster reaction of nitrobenzene (NB) for a mixture of 4-NP and NB.

Mu et al. [65] synthesized a series of CO₂-responsive microspheres with (PAM)/polydimethylamino propyl methacrylamide (PDMAPMA) interpenetrating (IPN) structures by reverse-phase suspension polymerization. With increasing DMAPMA concentration, the particle morphology transitioned from IPN to a core-shell structure. These particles can undergo reversible volume expansion and contraction through alternating treatment with CO₂ and N₂. In addition, non-agglomerated responsive particles of different compositions can be prepared by changing the cross-linking degree. The maximum response relative swelling volume (RSV) can reach 11.6% when the PDMAPMA loading is at 87%.

Qi et al. [66] used free radical copolymerization to create a responsive polyacrylonitrile-co-poly (N, N-diethylaminoethyl methacrylate) (PAN-co-PDEAEMA) copolymer, and then electrospun it into nanofiber membranes for selective oil-water separation. Nanofiber membranes have excellent properties such as large specific surface area and high porosity. When CO₂ was bubbled, the tertiary amine group was protonated, enhancing the hydrophilicity of PDEAEMA and causing molecular chain extension. Water can easily pass through the nanofiber membrane, while the oil is retained by the nanofiber membrane. In contrast, after the N₂ simulation, the tertiary amine group was deprotonated, the hydrophobicity of PDEAEMA was enhanced and the molecular chain collapsed, which effectively changed the wettability of the nanofiber membrane surface and achieved the purpose of oil-water separation.

3.1.3 Metallic Ions Crosslink Gel

HPAM [67] is widely used for profile control and water shutoff, mainly involving the formation of gels through coordination bonds between multivalent metal ions and carboxyl groups in HPAM. Common metal ions include Al³⁺, Fe³⁺, Cr³⁺, Zr⁴⁺, Ti⁴⁺, among others, and these multivalent ions can exist in the form of simple inorganic ions or soluble coordination compounds. The rate and extent of the reaction depend on the ligand within the crosslinking agent's molecular structure [68,69]. If carboxyl groups of metal ion complexes further bind with another carboxyl group on the same polymer chain, intramolecular crosslinking occurs.

Mo et al. [68] reported delaying the crosslinking of HPAM with Cr³⁺ by adding citric acid. This was effective because the ionization of HPAM is influenced by the pH of the system, and adjusting the pH of the buffering solution can control the release of CO₂, thereby delaying crosslinking.

Hadi Mosleh et al. [69] evaluated the PAM/Zr⁺ gel system under constant pressure of 50 psi CO₂ injection. The results showed that the polymer gel (polymer concentration: 1000 ppm and crosslinker concentration: 15 ppm) could reduce the CO₂ permeability by 99%. Hence, low pH may reduce the crosslinking rate and push the low-viscosity polymer slug into the remote well zone to achieve deep plugging, thereby increasing the CO₂ sweep efficiency.

3.1.4 Organic Crosslink Gel

Cross-linking mechanism of most organic crosslink gels is typically divided into two categories: One is the preparation of phenolic resin by condensation of polyhydroxyresorcinol. Further polycondensation between the phenolic resin and polyacrylamide can form a bulk polymer, thereby improving the stability and strength of the gel. The other is to form *in situ* gel with polyvinylimide (PEI) as a crosslinking agent under specific conditions. Among them, AM and monomers containing the tertiary amine group are used to form a gel *in situ* through free radical copolymerization [70,71].

Li et al. [70] reported a CO₂-responsive gel system with polyacrylamide as the main agent, and resorcinol as the crosslinking agent. CO₂ can create an acidic environment in the reservoir, leading to the release of formaldehyde from methenamine under acidic conditions. Polyhydroxymethyl resorcinol can be formed through the reaction between formaldehyde and resorcinol. The phenolic resin was formed via polyhydroxy resorcinol condensation. Further polycondensation between phenolic resin and polyacrylamide can form a bulk polymer, which improves the stability and strength of the gel. This effectively plugs highly permeable zones, improving CO₂ sweep efficiency and oil recovery.

Wu et al. [71] used AM and DMAPMA as monomers and PEI as a crosslinking agent to synthesize a delayed swelling CO₂-responsive hydrogel through free radical copolymerization, which contains tertiary amine group and crosslinking group (-NH). The hydrogel exhibited excellent swelling properties, expanding tenfold at pH ~4.5 and twofold at neutral pH ~7. Additionally, the gel remained stable for over 30 days at 80°C without dehydration, addressing CO₂ channeling issues.

3.2 Physical Crosslink Gel

Physical crosslink gel can also be called “supramolecular gels” or “self-assembled gels”, and are crucial soft materials with unique advantages. Unlike traditional chemical gels with covalent bond crosslinking, which form a network structure via inferior non-covalent interactions and have the property of reversible crosslinking [72]. Supramolecular gels are shape memory gels whose temporary shape is stabilized by reversible crosslinking. Based on the triggering mechanisms of the shape memory effect, it can be divided into two types: supramolecular interactions and dynamic covalent bonds. This kind of non-covalent bond interaction includes hydrogen bond interactions, π - π interactions, electrostatic interactions, van der Waals force interactions, dipole-dipole interactions, metal-ligand coordination, host-guest interactions, etc. [73–75]. They have found widespread applications in biomedicine, including drug delivery, gene transfection, protein delivery, biomedical imaging and diagnosis, tissue engineering, and biomimetic chemistry [76]. Self-assembly gels generally come in two forms: micromolecules self-assembly and macromolecules self-assembly [77].

The most significant feature of physical crosslinked gels is that their “sol-gel transition” is reversible under external environmental stimuli. This characteristic is particularly advantageous in the field of CO₂ plugging, allowing injection in a low-viscosity sol state that gels to block gas channels upon contact with CO₂. The broken gel can self-repair, making it a new generation of “smart” gel materials capable of automatically identifying and selectively plugging gas channels.

3.2.1 Micromolecule Self-Assembly

Some surfactant systems can form wormlike micelles (WLMs) by changing the critical micelle concentration (cmc), thereby undergoing gelation. This is the main mechanism of small molecule self-assembly gels. This category of surfactants includes cationic surfactants, anionic surfactants, non-ionic surfactants, different types of combined surfactants, zwitterionic surfactants, mixed systems of surfactants and auxiliaries, mixed systems of surfactants and solubilizing salts, Gemini surfactants,

oligo-surfactants, block copolymers, etc. [78]. Micromolecule self-assembly gels have the advantages of easy preparation, reversible and controllable rheology, economy, and environmental protection, and have been widely used in many fields.

Dai et al. [79] prepared a novel CO₂-responsive plugging channeling system by using small molecular amine compounds and a modified long-chain alkyl anionic surfactant. Before contacting CO₂, the system was a low-viscosity fluid and after contacting CO₂, the primary amine molecules were protonated, bridging two anionic surfactant molecules through electrostatic forces to self-assemble into WLM, resulting in a significant increase in viscosity and viscoelasticity. Rock core displacement experiments demonstrated a plugging efficiency exceeding 90%, effectively plugging CO₂ channels.

Shen et al. [80] selected a long-chain surfactant N, N-Dimethyl erucamide tertiary amine (DMETA) from 10 kinds of surfactants containing tertiary amine groups, it has a reversible CO₂/N₂ bubbling response. After CO₂ is bubbled, tertiary amine groups are protonated, the electrostatic repulsion is enhanced, and micelles self-assemble into a network structure. The viscosity of the system is increased to 605700 mPa·s, displaying viscoelastic behavior. This property enabled plugging high-permeability layers, improving sweep efficiency, and increasing crude oil recovery by 21.7%. When N₂ is bubbled, the system returns to neutral, the tertiary amine group deprotonated, and the viscosity eventually decreases. Thus, it can be used to address the issue of gas channeling during CO₂ flooding in low-permeability reservoirs. Plugging/unplugging can also be achieved via CO₂/N₂ bubbling.

Zhang et al. [81] synthesized a CO₂-responsive WLMs by mixing three different types of surfactants that containing N-erucamidopropyl-N, N'-dimethylamine (UC₂₂AMPM), sodium dodecyl sulfate (SDS) and N,N,N',N'-tetramethyl-1,3-propanediamine (TMPDA). When CO₂ is bubbled into an aqueous mixture, the tertiary amines in the TMPDA molecules are protonated. Through electrostatic attraction, "bridging" occurred between two SDS molecules, forming pseudo-gemini surfactants and creating a viscoelastic WLM. After CO₂ removal, the quaternary ammonium salts acting as spacer groups were deprotonated into tertiary amines, and the vermicular micelles of the pseudo-gemini surfactants were dissociated into spheroidal micelles of conventional SDS molecules with low viscosity. Ten years later, Luo et al. [82] first reported to use of UC₂₂AMPM to block CO₂ gas channeling for WAG through *in-situ* gelation. When N₂ is bubbled into the system, the system becomes neutral, and the tertiary amine group deprotonates, ultimately forming a low-viscosity emulsion. Compared to traditional WAG, the recovery rate is increased by 8.0%, proving effective in mitigating gas channeling.

Wang et al. [83] developed a switching foam using long-chain cationic surfactants N-sinapidepropyl-N and N-dimethylammonium bicarbonate (UC₂₂AMPM·H⁺) with carbon dioxide/ammonia water as an irritant. It acted as both a foaming agent and foam stabilizer. Under CO₂ conditions, the long-chain amine surfactant was protonated into cationic surfactant, promoting UC₂₂AMPM·H⁺ self-assembly into WLM. This increased the bulk phase viscosity and gas/liquid surface viscosity, enhancing foam stability. Upon adding NH₃·H₂O, the WLM network structure was disrupted, and the bulk phase viscosity and surface viscosity subsequently decreased and reducing foam stability. In addition, CO₂ can also act as a stimulus to protonate long-chain tertiary amine surfactants into cationic surfactants and form viscoelastic WLMs to stabilize the foam.

Gao et al. [84] prepared a novel CO₂-responsive zymphoteric WLMs by mixing N-octadecyl-N,N-dimethyl-3ammonio-2-hydroxy-1-propane-sulfonate (OHSB),N,N,2,2-tetramethyl-1,3-propanediamine (TADPA) and sodium salicylate (NaSal). The OHSB/NaSal/TADPA system shows morphological transformations between spherical micelles (a good flowing capacity) and WLMs (conducive to plugging) before and after purging CO₂. After CO₂ is bubbled into an aqueous mixture of these reactants, which will occur the reaction of CO₂-responsive mechanism one. On the one hand, it can

react with metal ions on the rock surface to precipitate for storage. On the other hand, the viscosity of the system is significantly increased with the increase of the intermolecular electrostatic repulsion, and the system is self-assembled into a three-dimensional network structure, making it effective in inhibiting gas channeling and increasing recovery.

Zhao et al. [85] prepared the surfactant erucic amide propyl dimethylamine (EAPD) as raw materials at a molar ratio of 1:1.1. In the absence of CO₂, EAPD molecules in solution exist mainly in the form of spherical micelles. When CO₂ is bubbled, EAPD molecules undergo a protonation reaction and become cationic molecules. With the introduction of counter ions, self-assembled aggregates are formed based on the synergistic effect of hydrophobic association and static electricity. It transforms the spherical micelle into a WLMs. Due to the formation of a three-dimensional network structure, the viscosity of the system increased significantly after CO₂ bubbling, and formed a gel with a certain strength. When encountering oil or N₂, the EAPD molecule undergoes a deprotonation reaction, and the WLMs turn into a spherical micelle again. Through mobility control ability plugging by the double pipes flooding experiments, the gel system can increase the recovery rate of low permeability reservoir by 18.7%.

Liu et al. [86] developed a double cross-linked frustrated Lewis pair network (FLPN) through a specific three-component reaction among triaryl borane, triarylphosphine, and CO₂. which is composed of permanent chemical crosslinks and dynamic CO₂ gas-bridged connections. The addition of CO₂ could adjust the density of supramolecular nodes in such FLPN, thus controlling the strength and toughness of the gel material. In addition, the self-healing of broken gels can be achieved by CO₂ stimulus through the reconstruction of dynamic covalent networks.

Zheng et al. [87] prepared a CO₂-switched, injectable, block copolymer hydrogel by the combination of coordination-driven self-assembly and stepwise post-assembly polymerization, star block copolymers (SBCPs) containing well-defined hexagonal metallacycles as cores. SBCP formed a stable single molecule micelle in an aqueous solution, in which the hydrophobic metal ring may be wrapped by the folded block copolymer chain. After CO₂ simulation, CO₂ and charged tertiary amines form water-soluble ammonium bicarbonate. The supramolecular amphiphilic polymer aggregates into large vesicles in aqueous solution through the hydrophilic/hydrophobic interaction. After N₂ simulation. The protonation of CO₂ with dimethylamino group is inferior, the hydrophilicity is reduced, and the vesicles are transformed into single molecule micelles.

Huang et al. [88] prepared a switchable pseudo-hydrophobic associative polymer using PEI and SDS. Under the stimulation of CO₂, the imide group of PEI is protonated, and produces electrostatic interaction and hydrophobic association with the SDS of the anion, thus forming a physical cross-linked network in the mixed aqueous solution, and the viscosity of the solution is significantly increased. However, as a result of the electrostatic interaction between negatively charged SDS and positively charged PEI, the hydrophilicity of PEI decreases, leading to the hydrophobic association physical crosslinked network contraction, and the solution precipitates white precipitates when PEI approaches neutral electricity. Continuous CO₂ bubbling increased the protonation degree of the amine group on the PEI molecular chain, enhancing the hydrophilicity of PEI and dissolving the precipitate, resulting in a significant increase in the solution viscosity of the SDS/PEI.

Yan et al. [89] reported a completely biobased pickering emulsion gel stabilized from modified cellulose (CNF), which was modified with a CO₂-responsive rosin-based surfactant, maleopimaric acid glycidyl methacrylate ester 3-dimethylaminopropylamine imide (MPAGN). As this surfactant has sensitive CO₂-responsive properties, allowing for switchable emulsification/demulsification, MPAGN can reversibly respond to CO₂ and N₂ between active cationic (MPAGNH⁺) and inactive nonionic (MPAGN).

Ali et al. [90] prepared a pH-responsive gel system by using cellulose nanocrystals (CNC) and imidazole salts to form a suspension. After the CO_2 is bubbled into a suspension, the pH of the system decreases, the generated carbonic acid dissociates a large number of protons, and through electrostatic interaction with the sulfonic acid groups on the surface of the imidazole salt, the cross-linking density of the internal network of the CNC hydrogel was enhanced, and *in-situ* gelation was achieved. Upon removal of CO_2 , the system's pH increased and the imidazole ions were deprotonated, the gel structure was destroyed and restored to the original sol, forming a tunable hydrogel by changing the concentration of imidazolium salt.

3.2.2 Macromolecule Self-Assembly

The CO_2 -responsive macromolecular self-assembly unit structure can be linear polymer or nonlinear polymer, or biological macromolecule, making the self-assembled of macromolecule more intricate than the self-assembled of micromolecule [77]. These macromolecules are typically divided into two categories: one with uncharged alkaline units (such as amine groups), which are hydrophilic at low pH and protonate into hydrophobic segments at high pH; and the other is macromolecules with acidic units (such as carboxyl groups), protonating into positively charged segments at low pH and carrying a negative charge at high pH. Common macromolecules with CO_2 responsiveness include polyacrylic acid (PAA), polyvinylpyridine (PVPy), polymethyl methacrylate (PMAA), PDMAEMA, PDEA, etc. [77]. As shown in Fig. 9.

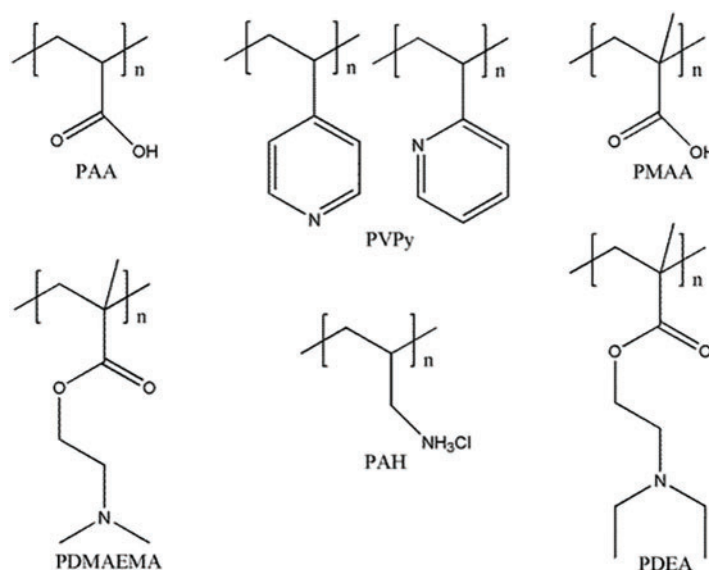


Figure 9: The common pH-responsive polymers (modified from Jiang et al. [77])

Luo et al. [91] synthesized a CO_2 -responsive triblock copolymer by one-step dispersion/emulsion polymerization. DMAEMA was used as a CO_2 -responsive monomer, 2,2'-azobis (2,4-dimethylglutaronitrile) (AVBN) and lauroyl peroxide (LPO) as initiator. By grafting PDMAEMA onto the triblock copolymer Pluronic F127, PEO_{100} - PPO_{65} - PEO_{100} for use to increase the viscosity of water in the WAG process. After CO_2 simulation, the viscosity of the aqueous phase increases significantly. The mechanism can be explained as follows: PDMAEMA is a weak base ($\text{pK}_a \sim 7.4$). Upon bubbling with CO_2 , the generated carbonic acid dissociated into a large number of protons, leading to the protonation of the tertiary amine groups in PDMAEMA. This caused the side chains to

extend due to electrostatic repulsion, resulting in the polymer having a larger hydrodynamic diameter, and increased entanglement of the polymer chains, thereby increasing solution viscosity. Upon N_2 simulation, the tertiary amine groups underwent deprotonation, restoring the solution to its initial state, and achieving CO_2/N_2 reversibility. Core flooding experiments demonstrated a 21.7% increase in conventional WAG oil recovery, effectively sealing CO_2 migration.

Younis et al. [92] synthesized carbon dioxide (H^+/CO_2)-sensitive poly (ethylene glycol)-b-poly (2-(diisopropylamino) ethyl methacrylate)-b-polystyrene triblock polymer (PEG-b-PDPA-b-PS) for programmed release of chemotherapeutic drugs against glioblastoma tumor. The release mechanism is as follows: the vesicle structure of multifunctional gold vesicles (GVND) first expands in the acidic microenvironment of the tumor, and then partially breaks through GVND under near-infrared (NIR) laser irradiation. Subsequently, the mild and high temperature generated by GV triggers the decomposition of NH_4HCO_3 to produce CO_2 . The *in-situ* CO_2 reacts with the tertiary amine group of PDPA, enhancing the hydrophilicity of the vesicles, disrupting its vesicle structure, and ultimately leading to the “bomb-like” release of doxorubicin (DOX) in the tumor tissues. This multi-stimulus-responsive programmed drug delivery and mild hyperthermia under near-infrared laser activation demonstrate potent antitumor efficacy and completely eradicated U87MG glioblastoma tumor.

4 Conclusion and Recommendations

Currently, the most relevant technologies involve measures and plugging agents for mitigating water flooding in heterogeneous reservoirs. It is evident that while CO_2 gas channeling and water channeling share similarities, there are significant differences as well. Therefore, the application of plugging agents used for water flooding control in CO_2 flooding presents considerable adaptability challenges and technical risks.

This paper, starting from the chemical response mechanism of CO_2 and the mechanism of gel channeling, thoroughly investigates the molecular structures, chemical compositions, response mechanisms, and response characteristics of CO_2 -responsive gels in various application fields, including double crosslink gel, metallic ions crosslink gel, organic crosslink gel, micromolecule self-assembly, and macromolecule self-assembly, aiming to apply them in CO_2 -EOR field. However, there are still the following deficiencies in the application of CO_2 -responsive gels in the development of oil and gas field:

1. The current application conditions only involve the gel performance under room temperature and pressure, which is greatly different from the use environment of CO_2 plugging channeling agents, and also limits the synthesis and application of CO_2 response plugging system to a certain extent, with certain limitations;
2. There are few studies on the influence of chemical response characteristics and reaction rate of CO_2 under different reservoir conditions;
3. There are few relevant studies on the effect of temperature and pressure on the phase change of supercritical CO_2 under reservoir conditions, and the effect of application technology on the formations and properties of gels;
4. The existing plugging agents have a high cost and are difficult to be widely used.

Therefore, developing low-cost and high-tolerance CO_2 -responsive gels is a crucial topic for solving various problems in current CO_2 -EOR technology. In-depth research on the relationship between structure and performance under reservoir conditions and the plugging mechanisms will be a major direction for future development.

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