A Perspective on the Prospect of Pickering Emulsion in Reservoir Conformance Control with Insight into the Influential Parameters and Characterization Techniques

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Abstract: In reservoir conformance control, polymer gels and foams are majorly used; however, they have drawbacks such as inducing formation damage, having weaker shear resistance, requiring a higher pumping rate, and limited penetration depth. Emulsions are a potential alternative that can address these issues, but they are not widely used. Current surfactant-based emulsions require high emulsifier concentrations for stability and often rely on multiple additives to address various factors, which makes the surfactant synthesis and utilization of emulsions quite challenging. However, Pickering emulsions, which utilize solid particles for emulsion stabilization, have emerged as a promising solution for reservoir conformance control. Compared to conventional polymer gels and foams, Pickering emulsions offer superior shear resistance, deeper penetration, and reduced formation damage. This review provides an overview of recent developments in the utilization of Pickering emulsions for conformance control, highlighting important parameters and characteristics that must be considered during the design and deployment of a Pickering emulsion for water shut-off operation. This review also sheds light on current challenges and provides recommendations for future development of the particle-stabilized colloid system.

Keywords: Pickering emulsion; conformance control; emulsion

1. Introduction

The recent Energy Outlook presents the demand for crude oil to grow up to 20–45% as the transition towards energy sustainability is reached by 2050 [1]. However, the current oil price scenario does not allow for the exploration of new reserves but rather the exploitation of untapped hydrocarbons from mature wells, which generally involves the implementation of recovery techniques such as water flooding and tertiary recovery methods, which are also complex and expansive.

Considering these aspects, it is necessary to develop and deploy inexpensive and environmentally friendly solutions to the field which utilize low dosages of injection fluids while getting maximum production out of the well. The water flooding operations, although quite effective, eventually, with time, require aid to block and divert the sweep flow from high permeable to low permeable zones where unproduced reserves are present.

Water production, along with hydrocarbons, is a common problem throughout the world. There are two possible causes for this matter. It can either be because water-producing zones, either natural or due to water flooding, do not allow oil to be catered to from low permeable zones. The other possible cause is the tendency of water to bypass hydrocarbons hindering the movement towards the production profile. This can occur

either right from the primary stages of the reservoir production or as a result of water flooding implementation. Water is produced at a much greater volume than hydrocarbons, and as a result, fluids reaching the surface are in the form of emulsion and therefore have to go through separation processes to attain the uncontaminated form. Groundwater treatment for utilization or disposal excessively increases the cost of production operations.

Conformance control refers to techniques that ensure the flow of reservoir fluids in a controlled and efficient manner throughout by maintaining the reservoir profile. These techniques can be physical-based for blocking or diverting fluid flow towards the targeted zone using bottom-hole tools such as screens, sleeves, and packers. However, mechanical means are expensive comparatively and, in certain cases, less effective. Therefore, chemical means are preferred, which can penetrate deeper zones and enhance the reservoir profile greatly by injecting fluids for enhancing the production profile via pressure maintenance or divergence towards lower permeability zones where the untapped hydrocarbons are present for recovery.

Chemical-based methods have been used for conformance control since the beginning of oil and gas drilling and production operations by using cement to block or divert zones that were prone to cause adverse effects in the reservoir, such as fluid loss in thief zones or productions that were no longer producing. With the passage of time, conformance materials such as latex rubber and silica gels have been employed for deeper conformance operations [2–6]; however, these were also not as efficient and generated formation damage. Eventually, this led to more efficient fluid systems such as polymer gels, foams, and emulsions.

Polymer gel-based plugging has been the conventional means of conformance for quite some time, followed up by foams. However, both have potential drawbacks, such as irreversible formation damage, lack of shear resistance, specific pumping requirements, and selective placement, which make their implementation far too costly and technically unfeasible [7]. Emulsions overcome these concerns but are rarely considered [8], providing comparatively better sweep efficiency, blockage, high shear resistance, ease of injection, and minimal formation damage. However, currently, surfactant-based emulsions are used, which require high emulsifier concentration for stability and multiple additives for different factors, thereby making synthesis and utilization a relatively difficult task.

Nevertheless, emulsions have gained prominence until very recently, especially with the readvent of Pickering emulsions, which has made their usage much more promising [9]. However, the work has mostly been on a laboratory scale and has not been applied to see the field-scale implications. Mostly the research being conducted considers in situ generation of emulsion, which in terms of laboratory scale, has proven to be successful but has potential drawbacks when applied in the field [10–13]. In this review, an effort has been made to summarize the current development in emulsifier technology and highlight the influential parameters that must be considered during the design, development, and deployment phases of an emulsion, particularly for Pickering emulsion for a well to attain successful conformance and also mention possible concerns that can arise by the implementation of micellar solutions instead of preformed emulsions based on the experience gained from other conformance agents and emulsion deployment in other operations. The databases used for this study are Scopus and Web of Science, using the keywords "emulsion" and "conformance control". The results obtained were screened through the respective database's advanced search filter tool and further by data analysis on MS Excel based on relevance to the focused research area. A total of 47 publications were selected and utilized to depict the current scenario in the area. This review can be used as a reference for Pickering emulsion utilization for conformance control operations.
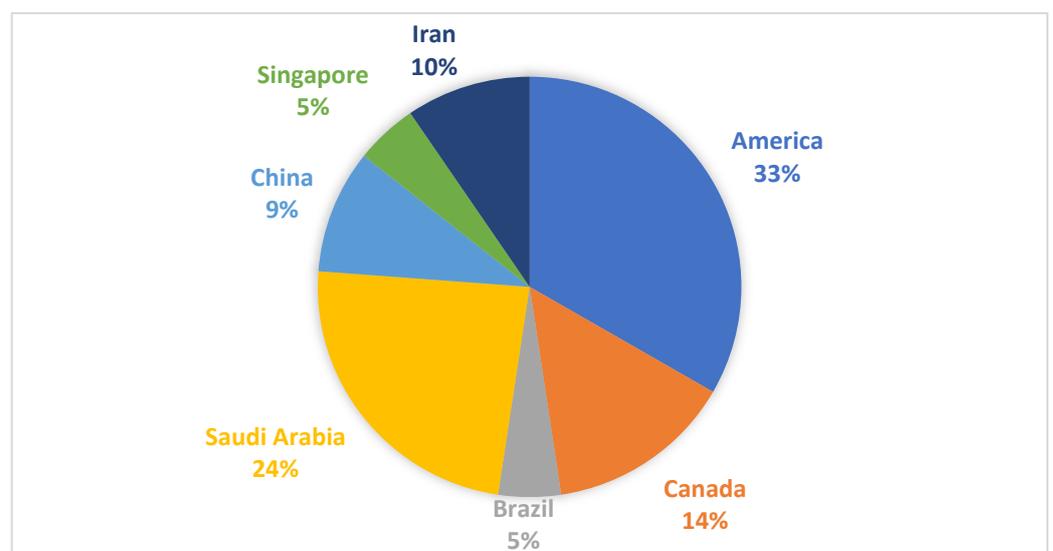
2. Emulsion for Conformance Control

Micellar solutions and emulsions were used during the early 1960s and 1970s for conformance control yielding promising results in the field but have since then been forgotten and are rarely considered for field-based implementation [10–12,14,15]. This is

major because of the advent of polymer gels for water shut-off, as evident from the graph trend in Figure 1. With their wide implementation, the drawbacks of polymer gels have also been identified, some of which have been addressed to an extent, such as susceptibility to high salinity and temperature conditions, yet some remain, as mentioned prior. Due to these concerns, along with the recent advancements in emulsifier/emulsion technology, there has been a rise in the development of emulsions for conformance control.
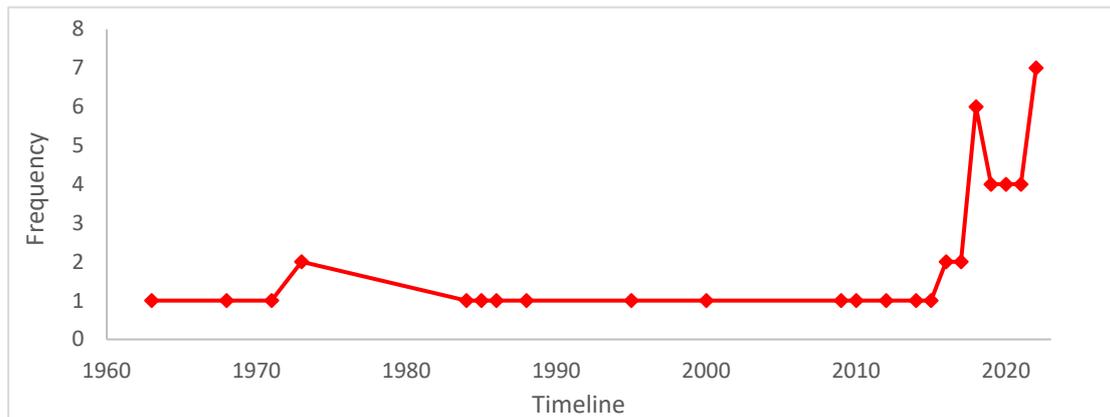
Figure 1. In total, there have been 47 publications throughout the 60 years related to the oscillating trend of emulsion utilization for conformance control; the fall is primarily due to the dominance of polymer gel in the field and the sudden rise due to the readvent of Pickering agents.

Emulsions have been applied vastly in enhanced oil recovery [16–18] and well stimulation operations [19–29] successfully and have shown desirable traits needed for conformance control; for instance, emulsified acids in well stimulation have successfully acted as good diverters as well by providing plugging effect in high permeable zones while moving fluid towards low permeable zones thus delivering satisfactory permeability enhancement as needed for both carbonate and noncarbonate rocks [21,28,30–32]. Figure 2 presents a trend regarding the publications coming from different regions on emulsion development for conformance control for heavy oil recovery, oil sands using steam-assisted gravity drainage and CO_2 injection.

Figure 2. The pie chart highlights a total of 22 publications on emulsion utilization for conformance control throughout the world (particularly for heavy oil recovery, oil sands using steam-assisted gravity drainage, and CO_2 injection).

Emulsions, in comparison to gels and foams, have the following: (i) better injectivity making them easier to pump, (ii) higher sweep efficiency and deeper penetration with a larger amount of uniform channel plugging, (iii) easier blockage removal along with minimum damage to the formation due to the Jamin effect as explained ahead in the mechanism of plugging in emulsion [13], (iv) higher oil recovery [33], (v) are relatively cheaper, And (vi) resistant to shear degradation. Due to the shear thinning ability of emulsion, degradation being less after the shear rate lowers, the viscosity returns, a trait not observed in gels or foams to this extent as in emulsions.

2.1. Type of Emulsions

An emulsion is classified on the basis of (i) the droplet size of the dispersed phase, (ii) the dispersion medium, and (iii) the type of emulsifier used.

i.   Emulsion type based on Droplet Size:

In terms of droplet size, emulsions are classified into macro-, micro-, and nanoemulsions. For conformance control, the droplet size mainly depends upon pore throat size as per the Jamin Effect rule, i.e., for effective plugging of droplet size > pore throat size as described by McAuliffe in his two papers [11,12]. The emulsions mostly considered for conformance control are coarse emulsions/macroemulsions having droplet sizes ranging between 1.5 and 100 micrometers while having a milky white color [34] or sometimes (rarely) microemulsion [35], which occupies a transparent or translucent shade (3 to 50 nanometer droplet size, or less than one micron) [36,37]. However, the results via microemulsion in [35] show hardly 53% water permeability (K_w) reduction.

ii.   Type on basis of Dispersion Medium:

The basis of the dispersion medium, an emulsion, especially a coarse emulsion, can either be water-in-oil (W/O) or oil-in-water (O/W), depending on which fluid is the external phase and which is internal. Microemulsions are kinetically sensitive to phases and can have three phases either an oil-in-water in oil (O/W/O) or a water-in-oil in water (W/O/W). For conformance control, O/W (oil-in-water) emulsion is selected more often; this has been discussed further ahead in the section on water–oil ratio [38].

iii.   Emulsion type on basis of Emulsifier:

Emulsifier is an integral component of the emulsion system; it combines the immiscible phases by lowering the interfacial tension between them. There are mainly two types of emulsifiers: (i) surfactants, which are liquid surface-active agents which alter the interfacial tension between two phases, and (ii) Pickering agents, which are solid particles that join the phases together with a very strong bonding by creating a barrier to prevent emulsion destabilization via accumulation of droplets. There is also the use of polymers as emulsifiers which make the continuous phase more viscous and provide a structural network to prevent coalescence [39].

2.2. Mechanism of Plugging in Emulsions

When an emulsion droplet enters a pore throat smaller than itself, it experiences a capillary resistance (to flow); this is because its leading (front) radius curvature is smaller than the back one, due to which pressure difference occurs. This effect is known as the Jamin Effect, as shown in Figure 3 [11,12,33]. Mostly it is considered necessary for effective plugging that droplet size should be greater than the pore throat [34], i.e., D.S. > P.T. = For effective Jamin Effect, where D.S. = droplet size and P.T. = pore throat. Therefore, when synthesizing an emulsion for plugging, moderate interfacial tension is kept so that the water phase provides adequate capillary pressure to plug oil droplets in the pore throat [40]. Moreover, this effect does not cause any formation damage like in the case of gels, where formation damage occurs due to the formation of filter cakes and precipitation of by-products [34]. Droplets are not only captured at the pore throat; their adsorption and capturing take place on the surface of the rock throughout [41]. This interaction depends

upon the emulsion's phase compatibility with the rock surface, which is a function of the polarity and viscosity of the emulsion; this affinity or repulsion between the rock and emulsion can either hinder or support droplet entrapment. There are three types of plugging mechanisms that can occur during the plugging process [42]. (1) Single droplet trapped at the pore throat, (2) multiple small droplets simultaneously trapped at the pore throat, and (3) plugging is caused by droplet adsorption.
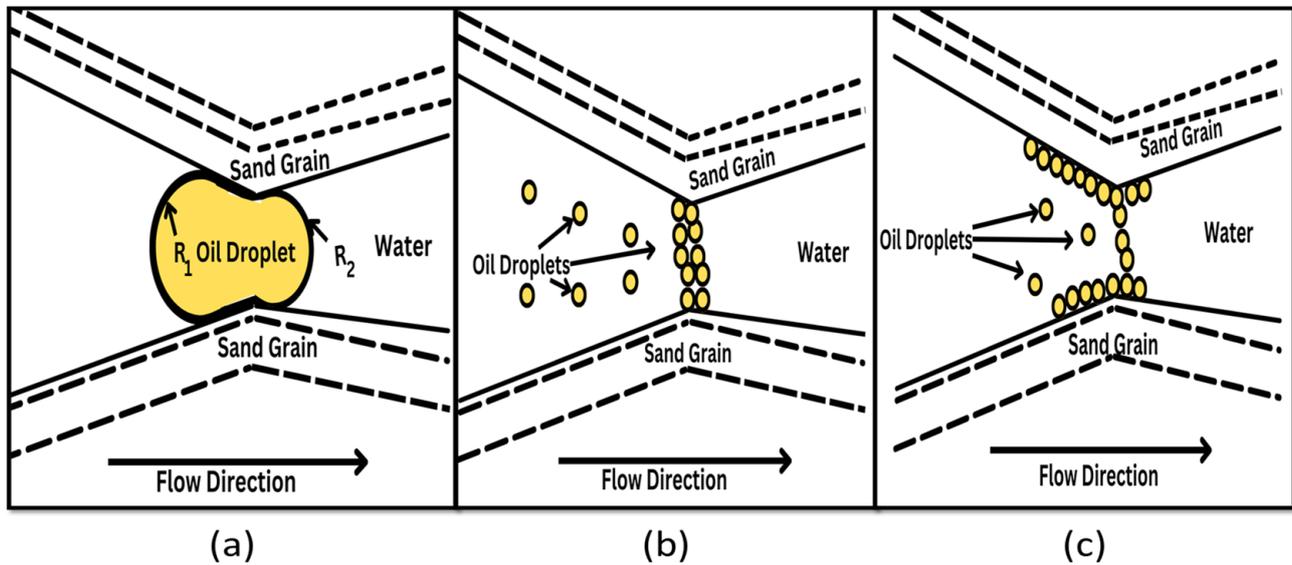
Figure 3. (**a**) Jamin Effect is the primary plugging mechanism of emulsion; for an oil-in-water emulsion, the oil droplet, upon entering the pore throat, experiences a capillary resistance due to the leading radius curvature being smaller than the back one resulting in a pressure difference needed for effective plugging. (**b**) Multiple droplets of varying sizes accumulate at the pore throat and block the passage; the droplet size heterogeneity allows for closing pore throats of different sizes. (**c**) Due to charge affinity between the Pickering particles and rock surface, droplet adherence occurs, thereby plugging the throat.

2.3. Drawback of Surfactants and Potential of Pickering Emulsifiers

Emulsions currently in use are mostly surfactant-based (molecular-surface active agents, liquids), which have short-term stability due to inefficiency to resist instabilities such as coalescence, flocculation, Ostwald ripening [43,44] and thus require high surfactant concentration. As well as being toxic, carcinogenic, allergic, and are not mostly biodegradable [45–47].

These drawbacks can be overcome by utilizing Pickering (solid particles) agents as a replacement. As these are known to be cheaper, more stable against instabilities, shear resistance, harsh temperature, and salinity conditions, have low particle usage, low toxicity, and are biocompatible and biodegradable. Figure 4 highlights the difference between surfactant-based and Pickering emulsions. Different types of Pickering particles can provide various other properties, for instance, conductivity, porosity, adsorption, etc. Due to these favorable characteristics, Pickering agents have been of wide interest in various fields, such as the food and pharmaceutical industries [45,46,48,49] as well as in petroleum exploration and production applications [50].

Table 1 highlights some of the recent developments made with emulsions in conformance control. It can be seen that the use of Pickering emulsions is becoming more prominent, and materials such as hydrophilic silica [51,52], cellulose nanocrystal [53], sodium montmorillonite [54], organoclay [55,56], and fly ash [57] have been tested out. However, in different cases, individual emulsifiers fail to provide the desired emulsification needed due to concerns such as degradation (especially in the case of surfactants and polymers), shear resistance, and wettability, which arise as a result of

harsh subsurface environmental conditions such as high temperature, pressure salinity, and pH. As a result, hybrid emulsifier-based emulsions are used, which utilize the combination of either surfactants, polymers, or particles to achieve a synergistic effect overcoming sole drawbacks of emulsifiers while using very minimum concentrations of each [51,52,58–60]. For instance, cationic followed by nonionic surfactants have been the most efficient in terms of stability and rheology when these have been mixed with hydrophilic silica and fly ash, both of which are negatively charged, thus providing strong bonding with cationic surfactants; however, these are susceptible emulsifier loss upon interaction with rock surface (sandstone are negatively charged) also are toxic. The better candidates are the nonionic surfactants which bond on the basis of hydrogen bonding; hence, there is no emulsifier loss concern relatively, and they have a higher viscosity than other ionic surfactants used to make them perfect for plugging. Moreover, these surfactants are nontoxic [51,52,57].
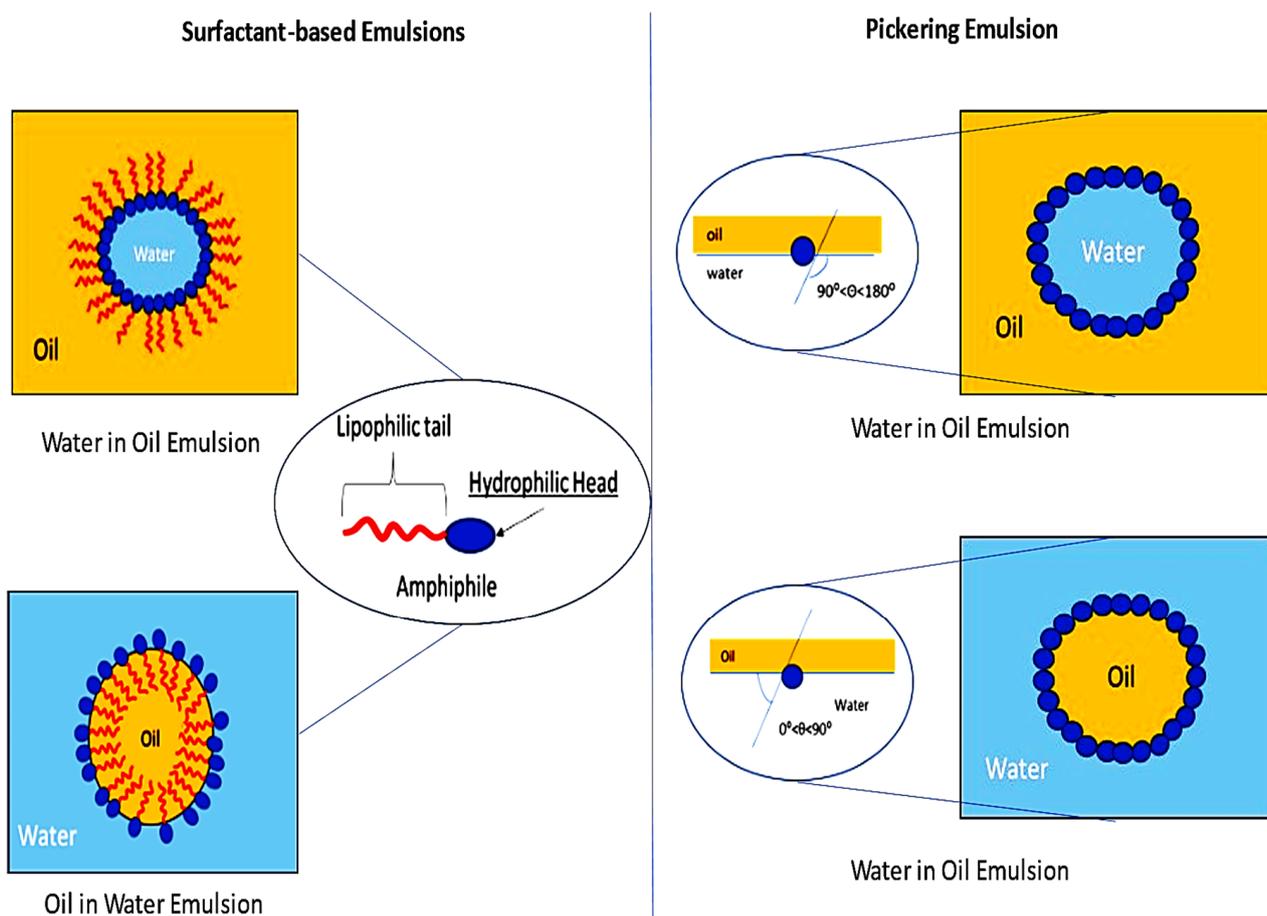
Figure 4. The illustration depicts the difference between Surfactant-based Emulsions and Pickering Emulsions. Surfactants consist of amphiphiles as their building blocks and the hydrophilic-lipophilic balance (HLB) value of a surfactant defines the phase.

Table 1. Recent Development in Surfactant and Pickering Emulsions.

S. No.	Author	Emulsion Type	Composition	Droplet Size	Formation	Outcome
1	Kim et al., 2017 [52]	O/W (Macroemulsion)	Oil Phase: Decane Aqueous Phase: Brine (8% NaCl and 2% CaCl$_2$) Emulsifier: Particle: Hydrophilic Silica (5 nm), Concentration = 0.01 wt% Surfactant: 4 types (Cationic, Nonionic, Zwitterionic, Anionic)	41–47 microns	Ottawa Sands	→ Surfactants which produced most stable emulsions are in the following order: Cationic > Nonionic > Zwitter ionic > Anionic → Emulsion possessed shear thinning behavior. Hybrid emulsions presented higher viscosity than those made solely with surfactants or particles. → Slight amount of NP aggregation due to high brine salinity prevents coalescence as particles form a network. → The ideal pH for sole NP emulsion formation is between 6 and 8; higher and rapid coalescence starts at lesser pH particles get neutralized due to large ionic volume in medium. → Nonionic surfactants are overall feasible, especially against surfactant loss than others.
2	Xu et al., 2017 [51]	O/W In situ Macroemulsion	Oil Phase: Decane Aqueous Phase: Deionized Water Emulsifier: Particle: Hydrophilic Silica (5 nm) 20 wt% diluted to 2 wt% Surfactant: Tween 40 (Nonionic Surfactant) (0.05 wt%)	190–210 microns	Bentheimer Sandstone replicated on glass	→ A microfluidic study was conducted in which the oil droplets moved towards the high permeable zones courtesy of NP adhered to O/W, forming a network and compressing in the high permeable zone while the aqueous phase sidelined into low permeable regions. This provided better blockage of high permeable zones and sweep efficiency of displacing fluid. Particle + surfactant makes a denser fluid than surfactant based.
3	Pandey et al., 2018 [53]	O/W (Macroemulsion) Preformed 50:50 ratio	Oil Phase: Dodecane Aqueous Phase: Water Emulsifier: Particle: CNC (250 nm, rod-shaped, treated with 12 M HCl)	7 microns	Sandpack	→ HCl-treated CNCs at 20 mg$_{CNC}$/mL$_o$ concentration were found to be most stable and had smallest droplet size; thus, continuous phase viscosity increased as a result creaming rate was slowed down. This is because of the excess particles, along with significant oil viscosity that formed a strong droplet network which provided effective blockage and robust stability. → Oil could enter emulsion-saturated zones at a pressure gradient 5× lower pressure gradient for water. → Compared with surfactant and polymer-based emulsions, which failed to block water flow, CNC-based Pickering emulsion performed effectively.
4	Chen et al., 2018 [33]	O/W Emulsion Ratio: 10:90	Oil Phase: Crude Oil (diluted with Kerosene for required Viscosity) Oil Viscosity for Core Flood was kept at 47.4 cp. Aqueous Phase Deionized Water + NaOH Surfactant: Dioctyl Sodium Sulfosuccinate	3–4 microns (avg 3.6 microns)	Sandpack	The following aspects were observed via core flooding: → Pressure drop increases (3.8 times) as oil viscosity increases. → Increase in oil viscosity increases emulsion viscosity, but emulsion viscosity reduces as injection rate rises. → Increase in oil viscosity leads to rise in frictional resistance, but too much increment in oil viscosity can cause increase in slipping behavior and change in path, which does not improve plugging efficiency.

Table 1. *Cont.*

S. No.	Author	Emulsion Type	Composition	Droplet Size	Formation	Outcome
5	Sadati et al., 2019 [61]	O/W Emulsion Optimum ratio: 20:80	Oil Phase: Intermediate Iranian Field Crude Oil Aqueous Phase: Synthesized Sea Water Emulsifier: Surfactant: Sodium dodecyl sulfate (HLB = 40)	—	Sandpack	→ Micelle solution of Sodium dodecyl sulfate and gasoil was injected to create in situ emulsion with water phase percentages (60–90% with 10% increments) into cores. → As observed from core flooding, emulsion injection resulted in incremental oil recovery up to 20%. → Emulsion with 80% water cut was technically and economically most feasible.
6	X. Zhou et al., 2020 [62]	In situ Emulsion EmulsionPolymer + Surfactant + CO_2 CO_2: Water Polymer Gel Ratio = 40:60	CO_2 Phase: CO_2 Aqueous Phase: Water Emulsifier: Surfactant: Polysaccharide linear polymer and a foaming agent	—	Carbonate	→ Two physical models were tested out: (1) Isolation of low permeable zone while opening the high permeable one. (2) CO_2 flooding followed by injection of a base gel into the cores while keeping both permeable zones open. → Model was more technically and economically feasible. Upon injection of 0.2 PV, it produced 46% incremental oi, while model 1 recovered merely 5% upon utilization of 0.4 PV of slug injection.
7	S. Li et al., 2020 [54]	O/W (Macroemulsion)	Oil Phase: Decane (5 vol.%) Aqueous Phase: Brine (10 wt% NaCl with 0.5 wt% clay particles in it) Emulsifier: Particle: Sodium Montmorillonite, (Clay particle dia. = 300 nm)	2.2–3 microns	Quartz Sand	→ Good long-term coalescence stability but poor creaming stability. → The clay-stabilized colloid system overall provided a 16% (67% to 84%) incremental oil recovery and mobilized all trapped oil in high saline conditions.
8	Saikia et al., 2020 [55]	W/O Emulsified In situ Polymeric Gel	Oil Phase: Diesel Oil Aqueous Phase: Field Mixing Water Emulsifier: Pickering Agent: Organoclay (Cloisite 20A) Polymers: (1) Polyacrylamide (3 wt.%) (2) Polyethyleneimine (1 wt.%)	1.485 microns	Berea Sandstone and Carbonate	→ Uniform penetration in both cases with unrestricted oil flow contrary to water flow. → After gel-based plugging, injections of Diesel oil followed by KCl flooding were conducted to check plugging. → The gel could withstand a differential pressure as high as 2000 psi. → In sandstone, Diesel Oil was only produced equal to the amount injected. → In carbonate, KCl brine found a channel to bypass plugged zones and got produced.
9	Pal et al., 2022 [35]	Microemulsion (ME), W1(O/W)-W3(O/ME/W)-W2(W/O)	Oil phase: Decane Aqueous phase: Deionized Water + Brine Emulsifier: Surfactant: SDS (sodium dodecyl sulfate, Anionic) Co-surfactant: Isopropanol	0.034 microns (Without salt) 0.138 microns (10,000 ppm) to 0.211 microns (80,000 ppm)	Sandstone	→ Microemulsion reduced 98% water cut by 30%, but with time (halfway through 0.84 PV of 1.15 PV), viscosity reduced as a result blocking efficiency decreased, and water cut increased to 53%. Showed slight inefficiency of the synthesized microemulsion.

Table 1. *Cont.*

S. No.	Author	Emulsion Type	Composition	Droplet Size	Formation	Outcome
10	Sun et al., 2022 [63]	W/O (Macroemulsion)	Oil Phase: Heavy Crude Oil of Xinjiang Oil Field Aqueous Phase: Formation Water varied from 20–90% at different salinities Emulsifier: Natural surfactants present in crude oil (Asphaltenes, resins, naphthenic acids)	0.2–4 microns with change from 20 to 90% in water content.	——	→ Emulsion non-Newtonian behavior (pseudoplastic/shear thinning). → Emulsion stability and DSD uniformity worsened with increase in water content. → Emulsion viscosity initially increased with water content from 20 to 40% but started decreasing after 40% to 90%. → Salinity had little impact on the stability of the emulsion due to naturally occurring surfactants (polar components) present in the heavy crude oil (used as the oil phase). → High viscosity allows for piston-like displacement due to high mobility control provided by high viscosity of the emulsion.
11	W. Wang et al., 2022 [57]	O/W (in situ micelle)	Oil phase: Kerosene Aqueous Phase: Brine (Salinity = 4263 mg/L), 0.5 wt% Emulsifier: Fly ash: Spherical and irregular, 150 nm (after milling) Surfactants: 0.2 wt% (Cationic, Anionic, and Nonionic)	——	Double-layered heterogeneous Sandpack Core	→ Stability was checked using a microscope and rheometer. The emulsion made from fly ash with cationic or nonionic were highly stable in comparison to surfactant and particle-based emulsions and worked effectively. → The cationic + fly ash emulsion improved oil recovery by 8.5% in intraformation heterogeneous cores. It was also used for sequestering the flyvash to the rock layers.
12	Mahboob et al., 2023 [56]	W/O Emulsified In situ Polymeric Gel O/W = 70:30	Oil Phase: Diesel Oil Aqueous Phase: Synthetic Sea Water and Formation Water Emulsifier: Pickering Agent: Organoclay (Cloisite 20 A) Polymer: Silica (28%) + NaCl (10%)	41.15 Microns	——	→ 10% NaCl and 28% Silica produce optimum gel. → Nonemulsified gel had a decrease in gelation time upon increase in salinity and temperature (100 °C), and upon increasing silica concentration, gel strength increased → Whereas emulsified gel had longer gelation time, although had good resistance at high temperatures and salinity. However, gel strength reduced with increase in silica concentration. → Bulk mixing produces direct emulsion (O/W), and drop-by-drop method generates inverse emulsion (W/O), → Optimized emulsified colloidal silica gel is made using drop-by-drop method.

2.4. Mechanisms of Pickering

There are three types of mechanisms that have been proposed: (i) mechanical (steric) barrier Mechanism, (ii) 3D viscoelastic particle network mechanism, and (iii) depletion interaction mechanism.

Mechanical barrier-based mechanism: The steric barrier is generated by the arrangement of adhering solid particles at the oil–water interface; also, this adherence of particles on the droplets generates repulsion between the droplets, thus preventing coalescence [64]. Moreover, the energy required for the detachment of particles from the interface is very high; thus, the stability is far more than the one provided by the surfactant mechanism [65]. As explained above, an effective Pickering agent has a three-phase contact angle close to 90°, at which the particle desorption energy required exceeds the thermal energy of Brownian motion; hence, an irreversible bonding is provided in Pickering emulsion particles that have partial wettability [66]. Particles that have a three-phase contact angle close to 0° or 180° are not considered Pickering agents, as the energy thermal energy of Brownian motion exceeds the particle desorption energy; that is, they tend to disperse [48].

Three-dimensional viscoelastic particle network mechanism: the interaction between three phases creates a 3D network that increases the viscosity of the continuous phase and prevents aggregation of droplets, thus preventing emulsion destabilization [50,67].

The depletion interaction mechanism: the 3D network is created by the addition of a nonadsorbing polymer which enhances bonding between particles and thereby aids in preventing the movement of droplets [68].

Out of the three theories, the mechanical barrier mechanism theory is supported the most [68–70]. However, in many aspects, the 3D viscoelastic particle network mechanism adds weightage, such as in the case when measuring the electrical conductivity of an emulsion to investigating the effect of oil volume fraction on emulsion and for determining the dispersion phase. The depletion theory is considered in circumstances when there is an addition of nonadsorbing polymer in the mix of the colloid system.

3. Influential Parameters Affecting Pickering Emulsion Design and Performance

Reservoir characteristics and fluid properties need to be considered during the design and selection phase to attain effective results.

3.1. Reservoir Heterogeneity and Permeability Ratio

Rock heterogeneity plays a major role in designing an emulsion since its stability, viscosity to mobility ratio, flow rate, and, thereby, blocking capability depends upon the level of pore size heterogeneity, type of permeable zones (fractured or unfractured), and their extent [71]. Similarly, the vertical and horizontal permeability contrast is a governing factor in the selection of emulsion injection either from the injector or producer well, while generally, injection from the production well is considered to achieve fast and relatively cost-effective blockage and avoid crossflows and undesired placement. In the case of low permeability contrast, the injector well placement gains more feasibility since the chances for crossflow of fluid into high permeable zones are less, whereas when the contrast is high near producer well placement is considered due to the generation of optimum pressure differential between high and low permeable zones resulting in effective blockage and diversion [72,73].

Before continuing further, it is worth highlighting a basic yet key parameter which is electrostatic interaction. This exists between the emulsion, the reservoir rock, and its fluids, as well as in between the different phases of the emulsion. This electrostatic interaction has an impact on emulsion properties such as droplet size, viscosity, stability, wettability, adsorption, and blockage capability of emulsion. The polarity, along with the molecular size and shape of each emulsion phase, influences its various other characteristics. For instance, in certain cases, the acid number or the amount of acid in oil due to polarity has a major effect on oil viscosity which affects the plugging performance of an emulsion [33,63]. In certain cases, the higher the acid number, the greater the viscosity [27,74,75].

3.2. Rock Surface Charge

Along with reservoir fluid polarity, the type and strength of rock surface charge help in deciding the type of emulsifier, oil, and aqueous phase to be used for a specific reservoir during conformance control. The allowable rate of adsorption and emulsifier loss needs to be considered, as these affect emulsion stability, precise (selective) placement, and blockage [76,77].

As per the scope of this study, a Pickering emulsion has two main traits, stability and wettability, which are influenced by multiple factors related to its three phases (oil, water, and solid particle), out of which the correspondingly significant ones are being discussed as follows: particle size, shape and charge, solid particle wettability and concentration, oil–water ratio, aqueous phase, salinity and pH, type of oil phase (i.e., viscosity and polarity of the oil phase), and temperature.

3.3. Salinity and pH of the Aqueous Phase

Changes in salinity and pH of the aqueous phase can reconfigure the particles, charge behavior, and can either lead to enhancement in stabilization or destabilize the emulsion. The change in ionic configuration can either lead to particles covering a larger interfacial area and providing vast dispersion of forces that would decrease droplet size and increase stability or cause defects in stability by drawing polar phases away from the particles due to yielding higher ionic strength [48,50].

3.4. Particle Characteristics

Pickering particle size, shape, and charge are the basic parameters that define its nature and, thereby, suitability for a specific application. The selection of a particle is very complicated when studied, and studies are being conducted to comprehend this aspect better. There is a correlation between the particle shape and charge, its concentration which influence its interaction among themselves and with the phases in the colloid system. In many cases, the same specification for a Pickering particle cannot be applied as in another due to the different nature of the aqueous and oil phases (droplet size, charge, concentration).

i.   Particle Size:

Generally, it is believed that the smaller the size of the particle smaller will be the droplet size which will result in better emulsification [78]. This aspect favors the fact that small particles are easily solubilized/dispersed in even insoluble solvents, thus, making them amphiphilic. This is because smaller particles tend to have a larger surface area per unit volume. This leads to better interfacial film formation and provides heterogeneous distribution of forces at the interface, which enhances stability. However, in certain cases, the particles are so tiny that they do not adhere to the interface effectively, nor do they create an effective barrier at the interface. For instance, Ge et al. [79] in a study, it was found that a moderate range (100–220 nm) of particles was needed for stabilizing an emulsion that could not be stabilized particles smaller or larger than this diameter range. Table 2 presents a few of the particles being used as Pickering agents for conformance control.

Table 2. Pickering Particles being used for conformance control.

Publications	Pickering Agent
[51,52]	Hydrophilic silica (5 nm)
[53]	CNC (250 nm, rod-shaped, treated with 12 M HCl)
[54]	Sodium montmorillonite, (clay particle dia. = 300 nm)
[55,56]	Organoclay, <10 μm
[57]	Fly ash, spherical and irregular, 150 nm (after milling)

ii.   Particle Charge:

Charge between particles has multiple ways of stabilizing the emulsion. The first mechanism is that of charged particles repelling one another and keeping the droplets dispersed. This is influenced by the capillary forces in between the particles; the capillary pressure induced helps in countering the gravitational forces, which tend to draw the particles closer to one another. Now because the capillary pressure leads to the formation of an interfacial film between the Pickering particles, this film consists of a thin layer of the continuous phase adsorbed onto the particles; this is due to affinity between the Pickering agent and continuous phase and will vary in each situation depending upon the nature of the three phases and the level of affinity between them. The capillary pressure due to repulsion between the particles helps in inducing the film as surface tension between the continuous phase and the particle increases. This interfacial film helps in keeping the particles at a moderate distance from one another to maintain the dispersion and, at the same time, in a proximity that tends to reduce the overall energy of the system that is keeping them dispersed. This is better known as steric repulsion [70]. The second mechanism is when the particles on the opposing droplets draw closer to one another and maintain a double-fortified barrier between the droplets; this is (as mentioned prior) due to the nature of the continuous phase and, to an extent, the dispersed medium as well. There is another mechanism involving enhancement in stability due to the presence of unadhered particles and the addition of polymers in between the adhered Pickering particles of opposing droplets. Both agents aid in increasing the structural integrity of the colloid system. This mechanism follows the depletion interaction mechanism theory mentioned above. The polarity and its intensity play a pivotal role in emulsion phase behavior and stability, which has been pointed out in the water–oil ratio section ahead.

iii.   Particle Shape:

Particle shape plays a vital role in the overall stability of the emulsion as the way the particles occupy the droplet area and distribute the forces around them. Previously it was believed that spherical particles provide better stability due to uniform surface interaction with the phases, whereas nonspherical would tend to have unsymmetrical interaction depending on the shape; the probability of achieving stability would differ as per the size and shape of the particle. However, recent development in investigating this aspect favors nonspherical-shaped particles over spherical particles. This is because irregular-shaped particles provide more complex and stronger interparticle interaction and heterogeneous forces distribution than that provided by regular shapes, creating a more effective barrier. Moreover, the capillary pressure between the adjacent particles is much stronger when their shapes are irregular or of different geometry [48,79]. As mentioned in the section discussing the difference between surfactant and Pickering emulsions, the stabilization energy for Pickering particle attachment to the water–oil interface depends upon the contact angle. Generally, the detachment energy required to separate the Pickering agent can be calculated using the following equation [80,81]:

$$E = \pi r^2 \, \gamma_{ow} \, (1 - |Cos\theta|)^2 \tag{1}$$

where E is the detachment energy, r is the radius of the particles at the oil–water interfaces, γ is the interfacial tension between the oil and water, and θ is the wetting contact angle. However, this equation is applicable to only spherical particle-based emulsions [68]. As the contact angle is very important for determining the energy value, and for nonspherical-shaped particles, the contact angle will vary. Moreover, the size and shape will define the dimensional changes in the equation. Therefore, it would have to be modified accordingly. For instance, for a rod-shaped particle, the equation will be modified as follows:

$$E = lb \, \gamma_{ow} \, (1 - |Cos\theta|) \tag{2}$$

where l and b are the length and width of the rod-like particles, respectively.

iv.   Wettability of solid particle:

The wettability of solid particles generally governs which type of emulsion (with respect to phase behavior) will be formed (i.e., W/O or O/W). As per the Bancroft rule and Finkle's empirical rule, the phase which wets the particle the most becomes the continuous phase of the Pickering emulsion [68,82,83]. Particles, which are hydrophilic, tend to have an affinity for polar medium, which is usually the aqueous phase meaning an oil-in-water emulsion. Whereas hydrophobic particles adhere to a nonpolar medium, thus producing water in oil. However, this parameter is best defined via the contact angle between the three phases. As mentioned above, for an effective emulsion, the Pickering particle must have a contact angle close to 90° [84,85]. There are further other parameters such as particle concentration, water–oil ratio, viscosity, and polarity of the phases, which also influence this factor which are discussed ahead.

v.   Particle Concentration:

The concentration of solid particles influences the size of the droplet and, thus, the stability of the emulsion. Just like surfactant-based emulsion, the concentration of the Pickering particle increases, the droplet size decreases, and the dispersion is more homogenous throughout the emulsion. As mentioned prior in the depletion interaction mechanism, the additional particles which do not adhere to the droplets provide structural integrity to emulsion stability. But there is a limit known as the critical micelle concentration (CMC) beyond which additional concentration of Pickering agents provides no change in the droplet size; the polarity of the particle might increase, which can perhaps fortify the emulsion phase stability [61,86–88].

vi.   Selection of Pickering Particle:

When synthesizing an emulsion, the above-mentioned factors should be considered during the screening process to attain an effective Pickering colloid system. Hence, from the literature review, the following points have been summarized for the selection of a Pickering particle.

As perceived since the discovery period, a Pickering emulsion for a particle is an effective Pickering agent. It must have three primary characteristics; only then will it be able to provide long-term stability and a robust emulsion. (1) It must be partially wettable in both phases. (2) It must possess a moderate surface charge that must be present for particles to adhere to the oil–water interface, (3) The particle size must be smaller than the required droplet size but not so small that it is not able to adhere to the droplet interface entirely. It should possess the required properties as per the need of the application (conductivity, absorbance porosity, degradability, etc.

3.5. Water–Oil Ratio

As mentioned, wettability is one of the critical parameters in maintaining specific phases of the emulsion. Along with the affinity/polarity strength of the particle, the water–oil ratio governs the discrete and continuous phases. Slight changes in a certain phase's ratio can change the affinity of the particle leading to a change in emulsion type; this also would depend upon how strong the affinity (polarity) of the particle towards a specific phase is, especially the original continuous phase. Moreover, the volume ratio also affects the viscosity of the colloid system, which as a result, has an effect on droplet generation, stability, and fluid flow. Different volume ratios of water and oil have been tried out 90:10, 80:20, 70:30, 60:40, 50:50, 30:70, 10:90, and 5:95, depending upon the type of oil used, mobility, and plugging requirement. For conformance control, O/W (oil-in-water) emulsion is preferred rather than W/O (water-in-oil), which is favored in well stimulation [38]. This is because O/W emulsion is moderately viscous like water which results in better injectivity and flow, whereas W/O has a higher viscosity than oil, thus causing difficulty in injection and generating a large pressure drop in the displacement profile [89]. Moreover, O/W emulsion has high apparent viscosity at higher salinity and are shear thinning [54,90] O/W emulsions also provides easier blockage removal and low

formation damage. An oil-in-water emulsion provides a wider range of plugging by control over droplet size, and due to the varying size of droplets in an emulsion, it is possible to plug heterogeneous pore throats in a formation [12,34,91,92].

Although some studies have tested W/O emulsion and have observed adequate results providing good mobility control with high oil displacement efficiency even in the field [13,63,93,94].

3.6. Oil Viscosity and Polarity

The selection of the type of oil and its volume fraction in the colloid system defines the performance of an emulsion in terms of stability, flow behavior, and plugging. Viscous oil should be taken for effective plugging due to an increase in frictional resistance but not too high, as oil droplets with very high viscosity show solid-like behavior, which can demulsify and cause formation damage. Moreover, after a certain increase in viscosity, there is no significant impact [33]. Highly viscous oil droplets tend to slow down the mobility of the droplet; thus, the liquid interfacial film cannot be formed, and particles do not adhere to it, leading to an unstable emulsion [95]. The results are the same in the case of W/O emulsions as well when the continuous being highly viscous tends to slow down the movement of the aqueous droplets and particles, creating a similar scenario. If the oil ratio is taken in a significantly lower amount (<50%) than the aqueous medium, then the viscosity of the emulsion is usually that of the aqueous medium, no matter how viscous the oil is, as it hinders its viscousness. The polarity of the oil phase, in some cases, tends to have a significant impact; generally, hydrocarbons are used as the oil phase in Pickering emulsion for the petroleum industry due to technical and economic feasibility. These are nonpolar in nature, and as mentioned in the wettability discussion above, nonpolar medium attracts hydrophobic particles. But in a case study, it was observed that even though the particles were hydrophobic, the nonpolar oils formed oil-in-water emulsions, whereas polar oil, such as vegetable oils, yielded W/O emulsions which lead to the importance of contact angle on adsorption behavior, which is influenced by the polarity, wettability, and ratio of the respective phases [87].

3.7. Temperature

Temperature is a physical parameter that can alter the viscosity of the phases as well as the particle adsorbing behavior which results in changes in droplet size and interfacial tension. This has an impact on emulsion stability similar to that of pH and salinity as it influences the ionic configuration of the colloid. For instance, during synthesis, high temperature causes the droplet to break further into smaller sizes, and ionic charges are excited due to the provision of thermal energy [29]. However, depending upon the particle and phase nature, change can occur during the propagation of emulsion in high temperature and high salinity environments where either this leads to stability or destabilization of emulsion. The phases at different temperatures can behave differently, such as evaporation of the aqueous medium or solidification of the oleic phase affecting overall emulsion stability and rheology [85]. Similarly, the gain or loss of thermal energy by the colloid system can influence the electrostatic interaction between the particles and the surrounding medium; for instance, high temperatures induce excitation in particles ions, causing an increase in Brownian motion, which leads to redistribution of particles at the droplet interface, change in adsorption rate at the oil–water interface or alter ionization of surface groups leading to either strongly packed, particle arrangement at the droplet interface reducing coalescence or loose distribution of particles at the interface having high affinity for one another or weak interaction with the phases thereby increasing chances of destabilization via coalescence [59,96–98].

4. Emulsion Characteristics and Measuring Techniques

Functional characteristics of an emulsion and characterization techniques have been discussed in the following section. Figure 5 and Table 3 depicts the interrelationship be-

tween the characteristics and the different characterization techniques along standards used.
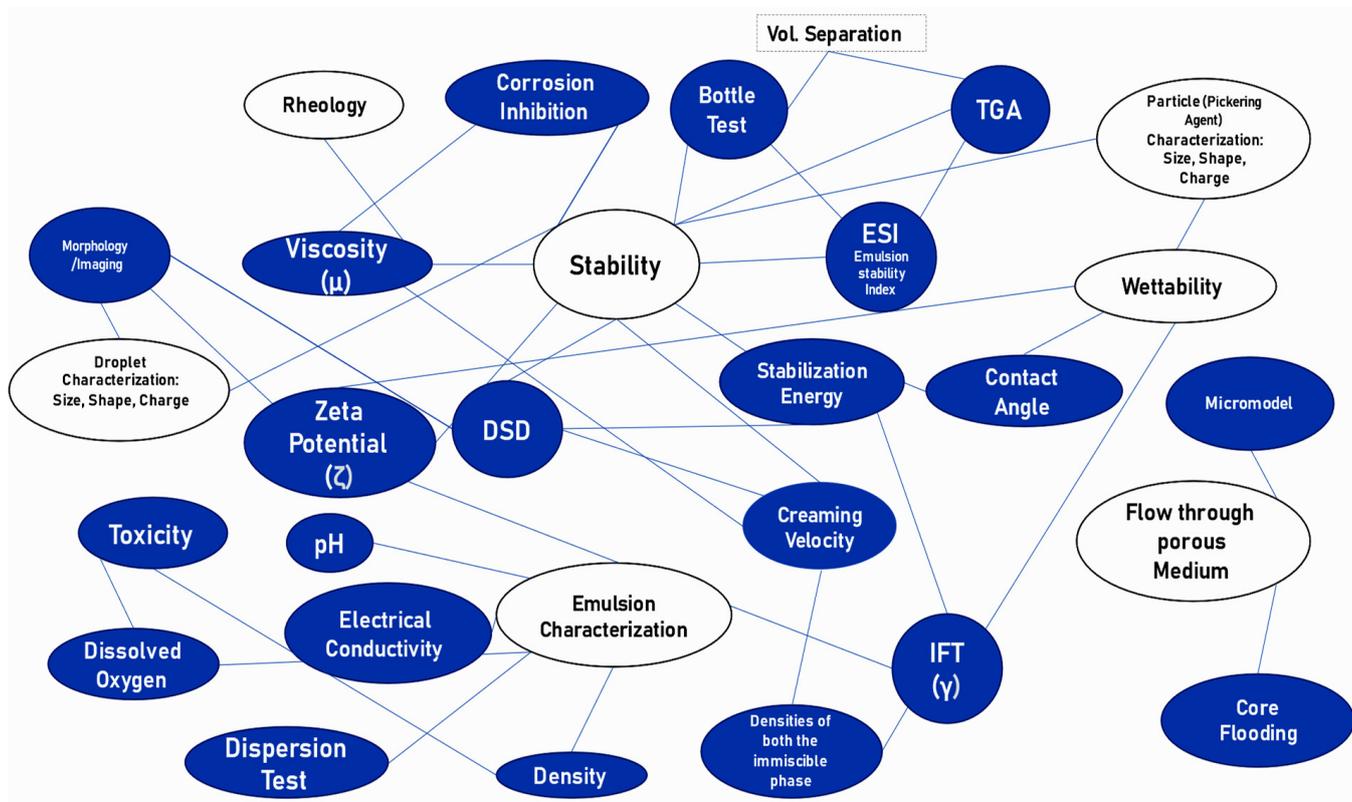
Figure 5. Parametric network shows interconnectivity among different emulsion characteristics, properties, and techniques, which are necessary for emulsion optimization during the design phase.

Table 3. Emulsion characteristics and their characterization techniques.

Characteristics	Techniques	Methods		Standards/Practices
Stability	Visual Observation	Bottle Test		---
	Thermal Analysis/ Mass Loss	TGA (Thermalgravimetric Analysis)		[99,100]
	Turbidity/Absorbance	Turbidity-based (Multiple Light Scattering)		[101]
	Spectrometry/Absorbance	UV-Vis Spectrophotometry		[102]
	Droplet Size Analysis	Ultrasonic Spectrometry (Acoustic-based)		[103]
		Light Scattering (Static/Dynamic)		[104]
	Surface Charge of Droplet	Zeta Potential	Electroacoustic-based	[105]
			Microelectrophoresis	[105]
	Imaging	Fluorescence Microscope		Best practices and tools for reporting reproducible fluorescence microscopy methods by Paula Montero Llopis [106]
		Cross Polarized Microscope		---
		Scanning Electron Microscope		[107]
		Field Emission Electron Microscope		
		Transmission Electron Microscope		[108]
Shear Thinning	Rheology	Rotational Rheometer		[109]

Table 3. *Cont.*

Characteristics	Techniques	Methods	Standards/Practices
Wettability	Contact Angle/ Interfacial Tension	Sessile Drop Method	[110]
		Pendant Drop Method	
		Spinning Drop Method (Modified Pendant Drop Method)	
		Du Nouy Ring Method	[111]
		Wilhelmy Plate Method	[112]
Flow through Porous Medium	Microfluidic Study Core Flooding	--- ---	--- [113,114]
Density	Oscillating U-tube Method		[115]
Total Acid Number	Titration Method		[116]
pH	Colorimetric Method		---
	Metal/Glass-electrode Method		[117]
	Semiconductor Sensor Method		[118]
Electrical Conductivity	Potentiometric method		[119]
Rock Porosity	Mercury Intrusion Porosimetry Gas Pycnometry		[120] ---
Rock Permeability	Steady State Method		[121]

4.1. Stability

A vital characteristic is that if an emulsion is not able to resist the physiochemical changes in its surroundings for a given period, the conformance operation cannot be successfully conducted up to the targeted zone. To ensure the formation of a stable emulsion, the following parameters are to be evaluated. Some of these parameters are direct measurements of stability for a given temperature and time, along with variations in salinity and pH.

i.   Bottle Test:

It is a very conventional and simple visual observation testing method based on gravity segregation/separation [122]. It helps in determining stability by scrutinizing the separation of emulsion for creaming or sedimentation with respect to time at different temperature and salinity conditions [26,55]. The extent of separation can be measured by noting down the thickness of the separating layer [123].

ii.   Emulsion Stability Index (ESI):

It is the measure of the emulsion's resistance against instability (coalescence and flocculation) over a specific period [124]. It is calculated to predict long-term stability. This is carried out by applying the volumetric method using the data related to volume separation obtained from the bottle test [55,125].

$$ESI = \left[1 - \frac{V_o}{V_e}\right] * 100 \tag{3}$$

where V_o = volume of oil separated from the emulsion and V_e = volume of the emulsion.

iii.   Imaging/Microscopy:

Microscopy helps in observing the dispersed droplets having a size smaller than 100 micrometers [126]. Droplet distribution and morphology (dimension and shape) play a pivotal role in identifying emulsion instability. Instability mechanisms such as flocculation, coalescence, and Ostwald ripening are all based upon the distribution and size of the droplets, and microscopic imaging techniques such as scanning electron microscopy (SEM) can help in detecting these processes and improving stability by preventing their occurrence

further during emulsion design/synthesis phase [123]. Combining microscopic imaging with a more precise technique, namely droplet size distribution (DSD), further strengthens surety regarding the stability of the emulsion. Electron microscopes such as field emission electron microscopes and transmission electron microscopes are the best choice for this purpose. The reason is due to its high resolution and ability to capture large surface area images quickly. Comparatively, the high resolution is most suitable for analyzing individual droplet shape, size, and morphology, as well as the distribution of droplets throughout the emulsion and at the interface [127,128].

iv.   Droplet Size Distribution (DSD):

An emulsion is targeted to possess homogeneous distribution of small-sized droplets with low variation so that the emulsion acts as a one-phase fluid system for a desired time. The heterogeneity in droplet size and level of dispersion indicates to which degree emulsion instability factors, such as creaming, flocculation, coalescence, etc., are likely to occur. For this purpose, droplet size and its distribution at different regions of the emulsion need to be evaluated. This allows for spotting the instability mechanisms which may be hindering the stability performance of the system. Emulsion stability is susceptible to salinity and temperature; therefore, during the design stage, emulsion needs to be tested for resistance against specific temperature and salinity ranges, which would be present in the subsurface. Generally, droplet size and distribution can be measured by the following techniques. As presented in Figure 6, most droplet size attained in published studies resides within the range of 1–4 microns; however, droplet size varies from reservoir to reservoir and depends upon the pore throat size, which also has heterogeneity in the formation, so variation in droplet size of emulsion helps in plugging pore throat of different size in a formation [12]. The droplet size is governed by the Pickering particle size, the viscosity and charge of the phases, the mode of dispersion, mixing speed, and temperature during synthesis as well as the reservoir conditions such as salinity, pH, and temperature.

Light Scattering Method: It utilizes a monochromatic beam of near-infrared light that travels through the emulsion sample. It provides the concentration and size of the droplets via the detection of the percentage and angle of backscattered light [129]. Higher concentration is indicated by the large percentage of backscattered light, while lower concentration by a low percentage. Meanwhile, the size of the particle is determined via its angular scattering and the intensity with which it scatters. Large particles scatter intensely at narrow angles, whereas small particles scatter intensely at wider angles. It is important to know that microscopic image analyses give number-based distribution, whereas particle size analysis techniques provide volume-based distribution. Although number-based distribution can be converted to volume-based, the accuracy of the particle size analyses provided is far more acceptable due to being measured directly [130]. In cases where it is necessary to provide a single point specification to represent droplet size distribution, then the median value should be presented as it is the most stable value generated by laser diffraction. The width of the distribution represents particles/droplets of a specific size range residing within a certain percentile in the emulsion system; this can be calculated by using Equation (4). Especially in conformance control, it is important to know droplet size heterogeneity as it helps in plugging multiple pore throats of diverse sizes [12].

$$Span = \frac{D_{90} - D_{10}}{D_{50}} \tag{4}$$

where *Span* represents the width of distribution, D_{10}, D_{50}, and D_{90} represent the distribution percentile of different particle sizes in the emulsion. If the span is not applicable, then two-point distribution should be represented, describing the coarsest and finest parts of the distribution (i.e., D_{90} and D_{10}) [130]. The light scattering technique (static/dynamic) can mostly measure sizes in the range of 0.3 nm to 5000 μm [131].
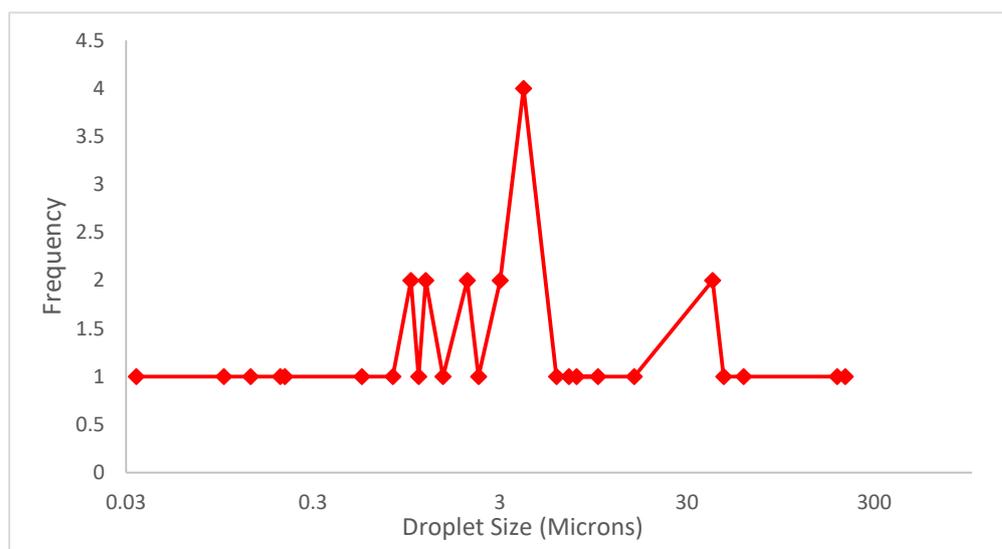
Figure 6. From 33 previous studies, the droplet size range resides between 1 micron and 4 microns, varying for each reservoir; the droplet size depends upon the Pickering particle size, viscosity and polarity of the phases, method of synthesis, and reservoir conditions (salinity and temperature).

Ultrasonic Spectrometry: It is a noninvasive technique that measures attenuation level in the frequency of ultrasonic velocity through which it calculates particle size distribution and concentration. This is because of variance experienced in the ultrasonic spectrum as waves travel through an emulsion, the velocity reduces, and the attenuation increases upon contact with the dispersed phase of the emulsion. Unlike the light scattering technique, which requires dilution of samples, causing a certain amount of compromise in accuracy, the acoustic-based allows for high particle concentration measurements due to the measuring mechanism relying on acoustics; it can measure even opaque samples. Generally, ultrasonic spectrometers can measure droplet size ranging from 5 nm to 1000 μm.

v. Zeta potential:

It is a property present in every droplet/particle dispersed in a colloid system. It helps in determining and optimizing the long-term stability of an emulsion without multiple trial formulations. The droplet inside a continuous phase experiences attractive and repulsive forces that are responsible for stabilizing an emulsion system or leading it toward flocculation and other instability mechanisms. The droplet is mainly influenced because of the emulsifier adhered to the droplet at the interface. This is explained in much more detail by the DVLO theory [132].

The droplets usually repel one another due to the dominance of the repulsive forces courtesy of the steric barrier created by the emulsifier; in the case of a Pickering emulsion, this keeps the emulsion stable. As discussed above, in the mechanical barrier mechanism, the particles are settled in between the oil–water interface with such high interfacial energy that to undo it would require a very high amount of thermal energy (like by increasing the temperature). If such an amount of thermal energy is provided, only then the attractive forces will be able to adhere to the particles and cause destabilization of the colloid (in this case, emulsion) via flocculation and coagulation. Apart from thermal energy increment to significant levels, other factors can lead towards adherence of particles. For instance, an increase in salinity in the surrounding can cause attractive forces to increase; in certain cases, the salt ion hydration phenomenon can occur, causing droplets to adhere towards the salts, thus reducing the electrostatic repulsion effect provided by the emulsifier to reduce (lose its dominance). Hence, if the attractive forces are moderate droplets, these overcome the slight repulsion in between them and start forming larger droplets leading towards coalescence. An ideal zeta potential value for a macroemulsion is around ±30 mV indicating a stable system. But the zeta potential value can be lower, and an emulsion can

still show resilient stability due to other factors such as the viscosity of the continuous phase, the interfacial tension between the phases, the presence of additional emulsifying agents, which can increase steric barrier and prevent coagulation [33,40,60].

Electrophoretic mobility and electroacoustic spectroscopy are the techniques that can be used for this purpose. Electrophoretic mobility is the movement of charged particles in an electric field. An electric field is applied to the dispersion and measures the velocity at which the particles move. The zeta potential can then be calculated from the measured velocity and the size of the particles. In comparison, electroacoustic spectroscopy measures zeta potential by introducing an ultrasound pulse to the dispersion, which causes movement in the droplets, thus creating an electric alternating current. This current is measured as a potential between two electrodes. The zeta potential is calculated from this measured potential. Electroacoustic spectroscopy does not require the emulsion sample to be diluted and can measure the zeta potential of (50%) concentrated samples [123].

4.2. Rheology

Emulsion flow properties are primarily associated with viscosity. It is a prominent characteristic when colloid stability is monitored as it reduces instability from occurring due to moderate fluidity and near homogeneous droplet dispersion in the system. Rheology is also important when considering the mobility/viscosity ratio of the emulsion system, which affects the mixing efficiency and differs in power requirement and pumping rate for pumping emulsion as per depth conditions of the well and specific applications (flooding, acidizing, fracturing, diverting/spacing, etc.). The shear resistance and flow behavior (shear thinning and thickening) are determined by using a rotational rheometer for an emulsion to be used for conformance control [123,133]. The plate geometry to be utilized depends upon the viscosity and stability of the emulsion. This is to account for the slipping behavior and evaporation tendency of an emulsion at high temperature and pressure conditions, which prevent accurate rheological characterization of an emulsion. For instance, a highly viscous sample can be measured with a parallel plate; however, for a low viscous fluid, a concentric cylinder is preferred. This is because, in a parallel plate, the wide surface area allows for higher chances of evaporation and slipping, whereas the concentric cylinder mitigates evaporation and slipping behavior by keeping the fluid at a raised level and within the confines of the cylinder. Moreover, it is recommended to have a large number of data points with a minimum spacing in between to capture the mechanical behavior of the emulsion accurately.

4.3. Wettability

It has significance on account of two factors: (i) phase behavior and (ii) stability of a Pickering emulsion. Contrary to surfactant-based emulsions where the emulsion phase behavior depends upon the hydrophilic-lipophilic balance of surfactant, in Pickering emulsions, wettability governs the phase behavior, whichever phase (oil or aqueous) wets the surface of the Pickering particle is the continuous phase; this is determined by measuring the contact angle. A contact angle close to 90° is preferred for a stable emulsion as it indicates partial wetting towards both phases and requires minimum energy for maintaining stability. To measure contact angle and interfacial tension, generally optical and force tensiometers are used.

Optical tensiometers comprise a syringe-like dispenser, a sample stage, and a light source for highlighting the emulsion droplet on the sample stage. Using the sessile drop method, which measures the contact angle as the droplet is dispensed/placed on the solid surface [134,135]. If the droplet spreads, the angle will be small, meaning it is hydrophilic in nature, whereas if the droplet forms a bead-like structure upon the solid surface, the angle will be large, highlighting lipophilic affinity [136]. Thus, depicting the emulsions phase behavior, whether which phase is continuous if as the mentioned angle is less than 90° hydrophilic nature is prominent meaning water is the continuous medium, whereas if greater than 90° then vice versa. If an angle close to 90° is achieved, then a stable emulsion

is obtained having an affinity towards both phases with a strong interfacial bonding due to the minimum energy required to do so. The interfacial energy at this position, as mentioned in the mechanical steric barrier mechanism, is very high. This does not only tell the emulsion's phase behavior but can also help in determining the hydrophilic/phobic nature of the solid rock surface as well as its surface free energy, which is measured by using the contact angle value. Therefore, it is best to also evaluate the interaction between the emulsion and rock surface using a solid etched layer of the core sample.

Moreover, this leads to the attainment of moderate interfacial tension, which provides an effective Jamin effect. Low interfacial tension cannot provide sufficient capillary pressure, while high interfacial tension will lead to emulsion instability and droplet breakup at the pore throat. Therefore, moderate interfacial tension must be attained so that the water phase gives enough capillary pressure to plug the oil droplet in the pore throat.

In certain cases, the interfacial tension measurements between the three phases need to be made on a very minute level. A spinning drop tensiometer can be utilized, which utilizes a modified form of pendant/rising drop method. The droplet is dispensed in a mixture of the Pickering agent, and the continuous phase is held in a capillary tube which is then rotated at specific speeds. Inside the tube, the centrifugal forces will push the continuous (denser) phase outwards while the droplet (less dense) is pushed inwards (towards the rotational axis). As a result, the droplet starts deforming while its interfacial area increases. The interfacial tension counteracts the area increase. This phenomenon helps in determining interfacial tension by analyzing the drop shape. This is measured when equilibrium is reached, and there is no change in the shape of the drop shape for a set time [33,137].

The force tensiometer comprises a probe (du Noüy ring or Wilhelmy plate) attached to a highly sensitive balance; the probe is displaced and pulled back from the sample at a controlled rate. It measures interfacial tension by measurement of the force exerted upon the probe at the liquid-liquid interface. The size, shape of the probe, the contact angle between the probe and liquid interface, and the interfacial tension at the liquid–liquid interface govern the force measurements. Generally, the Wilhelmy plate is preferred for highly viscous liquids, while du Noüy ring is considered for low viscous fluids [138].

4.4. Flow through Porous Medium

i.   Microfluidic Study:

Micromodels are ideal for studying rock morphology and flow behavior at the pore scale, providing much more clarity on essential information than attained on a macroscale. Micromodels are also known as a lab-on-chip setup. It is comparatively less costly than core flooding experiments utilizing droplets of emulsion for experimentation while providing a significant amount of information. Usually, these models are made from materials such as glass, silicon, and polydimethylsiloxane (PDMS). However, geochemical processes such as adsorption behavior during flow cannot be observed with such materials, which is much needed to understand the blocking phenomenon in conformance control. Recently rock wafers, as well as rock coating onto PDMS, have been used for micromodels in order to evaluate reactive transport in porous media [139,140]. This method is most suitable for analyzing sweep efficiency and pore throat blocking capability of an emulsion at a minute scale. High-speed microscopic cameras are utilized with micromodels to capture static and dynamic parameters. This has allowed us to study properties such as emulsion stability in dynamic conditions (at steady state), which is considered much more feasible than static stability tests [51]. The type of plugging mechanism in place, along with the adsorption tendency of the droplet and emulsifier on to rock surface, are examined on a small scale at a low cost.

ii.   Core Flooding:

The core flooding is the true performance evaluator of an emulsion at the laboratory scale to act as an effective conformance agent at reservoir conditions [54,57,63] before

field deployment. The treatment helps in understanding fluid transport and blocking mechanism and rate of change in permeability before and after conformance agent injection. Optimized treatment design can be developed for varying reservoir conditions by testing the emulsion composition, phase concentration, and injection rates. This, in combination with a microfluidic study, can provide a better understanding of adsorption and flow behavior, droplet entrapment/plugging mechanism, and the effect on recovery efficiency. For conformance control, usually parallel core setup is carried out [12], or double-layered heterogeneous sandpack cores are used [57].

4.5. Basic Characterization

For conformance control, emulsion rheology should have a robust shear resistance (high elastic modulus/storage capacity/stability), moderate mobility, a viscosity that can provide good sweep efficiency (divergence), and effective plugging capability as per the specific formation's physical properties (degree of heterogeneity in porosity and permeability, wettability) and implementation depth (if near the wellbore, deeper propagation is needed). To achieve a successful design of an emulsion with the above-mentioned conditions in mind, several factors which influence colloids phase and flow behavior need consideration, such as the viscosity and density, chemical composition, pH, total acid number, the electrical conductivity of both the phases with respect to which mainly the oil/water volume ratio is designated. The size, shape, and distribution dispersed phase (discussed above), as well as the shape, charge, and size of the Pickering particle, should be known prior for a better selection of chemicals and their respective concentrations during synthesis [39].

i.   Density:

The density of the emulsion, as well as of the immiscible phases, must be known. The density of the aqueous and oleic phases is primarily important for determining the creaming velocity of an emulsion, as shown in Equation (5).

$$V_{Stokes} = (2gr^2(\rho_2 - \rho_1))/(9 \times \eta) \tag{5}$$

where g is the acceleration due to gravity, r is the radius of the droplet, ρ_1 and ρ_2 are densities of the two immiscible phases, and η is the shear emulsion viscosity. This equation is based on Stoke's Law, which mentions how the stability of an emulsion can be measured by observing the creaming velocity of the emulsion droplet, either rising or falling (i.e., creaming or sedimentation). The emulsions with high resistance to this phenomenon tend to possess good stability as this indicates a colloid system having a relatively homogeneous distribution of droplets dispersed evenly. Therefore, knowing the densities of each of the phases in the system allows us to design the colloid better, as the influence of characteristics of one phase can greatly impact colloid stability and performance. The concentration of the respective phases in the colloid can be changed accordingly to reduce these adverse effects as much as possible.

ii.   Viscosity:

The viscosity of individual phases is a knowledge that helps in accessing the quantity of each phase as per the need of the application. For instance, the frictional resistance and overall viscosity of the emulsion increase as oil viscosity increases; however, if the dispersed oil volume fraction is in small amounts, then the viscosity of the emulsion is predominantly that of the aqueous [12,33]. Moreover, the highly viscous oil tends to provide better plugging performance which is also dependent upon the wettability and roughness of the rock surface as well as on the adsorption characteristic of the emulsion. Therefore, to attain effective sweep, plugging efficiency, and stability, the desirable mobility ratio will be based upon the respective phase viscosities as that governs the volume fraction of each of the fluids in the emulsion.

iii.   Total acid number:

The total acid number (TAN) tends to increase the viscosity of a fluid, as the acidic content can cause them to become strongly bonded/associated, causing an enhancement in viscosity (resistance to flow). This tends to mostly be a favorable property, especially when highly viscous oil is preferred, as is in the case of conformance control (explained above in the viscosity section). This can also have adverse effects in terms of an increase in the corrosiveness of the fluid. Hydrocarbon-derived oil (such as petrol-based diesel) tends to, while possessing a high viscosity, have a low acid number due to the presence of rarely any olefinic content (<0.5 mg of KOH/g); this puts it at a technically feasible position due to being less corrosive [74]. Whereas triglyceride oils comparatively have a higher risk of inducing corrosion due to high levels of acid value (e.g., jatropha oil acid value = 32.8 mg of KOH/g), especially at high temperatures [27,141,142]. Therefore, the TAN of each fluid phase must be determined to foresee any harmful effects the fluid might produce later.

iv.   pH:

Wettability (adsorption) and rheological behavior are highly influenced by the pH value of the emulsion and its respective individual phases. The interaction of emulsion with the mineralogy of the rock can affect the plugging performance of the emulsion as the slipping behavior and frictional resistance are influenced by the charge–charge interaction between the liquid–¬liquid and liquid¬–solid interfaces; this is governed by the pH value [33].

v.   Electrical Conductivity:

It is carried out to evaluate the influence of oil fraction on the emulsion. As the oil percentage increases, the electrical conductivity of the emulsion decreases; this also corresponds to the severity factor of the emulsion, i.e., the more severe the emulsification (viscous), the lower will be the electrical conductivity [143]. This is mostly because of slowness in the fluidity of the droplets if oil is a continuous phase as per the 3D viscoelastic theory, and the possibility can arise vice versa in the dull movement of oil droplets in a continuous aqueous phase. In O/W emulsions, it is carried out to check the severity of emulsification and the influence of oil volume fraction on the emulsion, whereas generally, in W/O, it is carried out to validate the formation of oil as the external phase [55]. This helps in validating resistance to instability as a moderately viscous emulsion lowers the chances of flocculation or any other defects.

5. In Situ vs. Preformed Emulsion

The majority of the research conducted on emulsion development for conformance control has been on a laboratory scale; only a rare field application has been reported. Mostly, laboratory publications have adapted micelle injection/in situ means of emulsion. There are drawbacks to this technique in comparison to preformed emulsion injection; firstly, laboratory trials relatively cannot account for the deep propagation of the conformance agent adequately. This is prominent from the vast use of polymer gels on a field scale where preformed polymer gels have been favored over in situ gels [144,145] as well as from the use of emulsion in other exploration and production applications such as well stimulation [29,146–152]. There have been reports of preformed emulsions outperforming in situ emulsions in blockage performance [94].

Micelle is not truly a one-phase fluid and is sensitive to changes; thus, it will have chromatographic fractionation concerns as each component will have a separate traveling time and adsorption tendencies, leading to emulsifier loss upon interaction with rock surface, partitioning tendencies, susceptibility to contamination from reservoir mineralogy, high heterogeneity in droplet size, and higher chances of crossflow and unselective placement. Therefore, it can be hard to deploy and maintain the injection pressure due to the highly responsive nature of the micelle. Preformed emulsion, on the other hand, is relatively robust to the reservoir conditions and possesses shear thinning rheology viscosity and mobility ratio can be optimized and provide great sweep efficiency with the accurate

selective placement of the emulsion on the blockage zone with minimum concerns over pumping requirements.

6. Micro vs. Macroemulsion

Microemulsions utilize large amounts of surfactant concentration, making them expansive. Although microemulsions are thermally stable but are sensitive to changes in fluid content and salinity, this can alter the fluid behavior and emulsion type on the basis of dispersion medium [18,35]; this is due to having a high interfacial area. This can affect their plugging efficiency. Macroemulsions are relatively cost-efficient, more robust to changes in fluid volume because of their comparatively smaller interfacial area, and have relatively high viscosity making them more suitable for plugging [33,153]. As per the literature review, macroemulsions are favored over microemulsions; based on droplet size, microemulsions account for 28%, while macroemulsions are vastly developed on a laboratory scale.

7. Conclusions

Emulsions have rarely been used for conformance control operations despite the beneficial characteristic of the colloid system. It has been overshadowed by the vast usage of polymer gels. The emergence of solid particles as emulsifiers has increased the chances for emulsion to be considered as a suitable candidate to replace the conventional gel-based system. In this review, the potential of Pickering emulsion has been highlighted along with the recent strides made in the area and the current limitations. Most importantly, the influential parameters and techniques to evaluate the effectiveness of an emulsion have been reviewed to provide guidance for new researchers in the field.

Pickering emulsions provide better stability and plugging at relatively low concentrations, along with being ecofriendly. With the selection of a suitable Pickering agent and thorough evaluation at the laboratory scale, while keeping influential parameters and characteristics into consideration during the design phase, Pickering emulsion can soon be utilized in the field widely. Pickering emulsions have shown the potential to act as efficient conformance agents. However, the current Pickering particle [51–54,57], although relatively better than surfactant-based emulsions and gels, lacks in certain areas as per the technical, economic, and current environmental concerns such as self-emulsifying stability, susceptibility to degradation under high temperature and salinity conditions, limited availability of the material, and difficult to dispose of. These can be improved by methods such as functionalizing Pickering particles with carboxylic functional groups to provide higher hydrophilic affinity and resistance to salinity and the use of organic waste to synthesize Pickering particles, which can reduce cost and produce a more ecofriendly product. The recent use of carbon dots as colloid stabilizers [9] has shown promising results in Pickering emulsion for drug delivery [154] as well as for foam-based enhanced oil recovery [155]; this creates possibilities for its use in developing emulsion with desirable qualities for conformance control especially carbon dots synthesized from organic waste material [156].

The deployment of emulsion is another area where the focus should be made to ensure effective placement; for this purpose, laboratory-scale flooding tests should be conducted on cores of 8 inches to 12 inches, and field-based evaluation should be increased to review the prominent drawbacks in deployment design through results from diverse injection operation conditions at various fields and improve upon them as carried out with polymer gels. This will allow conformation on the use of in situ or preformed emulsion as well as the specific type of emulsion to be utilized. Pickering emulsion can be considered as the next generation conformance agent considering the rapid pace in their development and might soon replace other conformance agents due to the technical, economic, and environmental benefits it provides.

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