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Utilization of Locally Formulated Chemical Demulsifier for Crude Oil Demulsification: A Review

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ABSTRACT

Emulsion is a critical problem in the oil and gas industry, from production through refining. It must be prevented or treated by the oilfield operator to ensure that the crude oil meets API standards. Emulsions can be oil-in-water, water-in-oil, or multiple emulsions, and require crude oil, water, an emulsifying agent, and emulsifying conditions. Several approaches, including mechanical, thermal, electrical, and/or chemical, are used to manage emulsions in the oil and gas industry. Still, the chemical approach is most widely used due to its technical, economic, and environmental viability. The chemical demulsification technique involves adding chemicals to break the bonds between brine and crude oil, using mechanisms such as flocculation, coalescence, and sedimentation. Several demulsifiers are available, but these chemicals are expensive for developing countries and are not environmentally friendly. In this study, a comprehensive review of the demulsification performance of locally formulated demulsifiers was conducted. Based on the study's results, a comprehensive understanding of the emulsion formation concept, mechanism, and types is necessary for designing a suitable demulsifier. Nicotiana Tabacum recorded 73.33% efficiency, orange peel recorded 60% efficiency, while soya bean husk oil recorded 45% efficiency in demulsification. Camphor, Vegetable Oil, Alum, and Liquid Soap were identified as key ingredients in the formulation of a local demulsifier, each playing a distinct role. Speed and Temperature increase the demulsification of locally formulated demulsifiers. The demulsifier performance of the locally formulated demulsifiers can be optimized using software.

Introduction

Crude Oil, by default, exists with natural gas and brine water in the reservoir pores and is produced together through the wellbore. While flowing from the wellbore to the surface, these immiscible fluids are temporarily emulsified by a continuous mechanism of shear reduction at valves, chokes, and the wellhead, and pressure [1]. Crude oil-in-water emulsions are relatively difficult to manage because they are stabilized by naturally occurring surface-active agents [2, 3]. The water within the crude oil poses various obstacles in the downstream sector, such as damage to process facilities, corrosion of pipelines, pumps, and the downstream distillation column, and increased associated costs [4]. There are many methods for removing water from emulsions, both industrial and research. Emulsion is a critical hurdle in the oil and gas industry, from production through refining. In most cases, oilfield operators choose to prevent emulsion

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formation or treat already-formed emulsions, depending on environmental requirements and cost. This involves mechanical, thermal, electrical, and/or chemical approaches [5, 6].

Concept of Emulsion

An emulsion is a colloidal phase formed due to the distribution of an interior phase (dispersed phase) in an exterior phase (continuous phase). The interior phase drapes the exterior phase due to its resistance to separation and coalescence, and this is stabilized by an emulsifying agent at their interface. Crude oil is a complex hydrocarbon fluid comprising napthenic acid, resins, and asphaltene, with asphaltenes being responsible for the increased stability of water-in-oil-emulsion, which is present in crude oil [7]. Crude oil is grouped into 4 major fractions using SARA (saturates, aromatics, resins, and asphaltenes) analysis based on its polarity and solubility in solvent. Table 1 shows the component of a typical emulsion and their corresponding description.

Table 1. Component of a typical emulsion

S/N	Component	Description	Ref.
1	Asphaltenes	Dark brown amorphous granular substance Specific gravity above 1 Represent hexane or pentane in the insoluble portion of the oil Decomposes at temperatures beyond 300-400 ºC Influenced by temperature, pressure, and oil composition variation	[8, 9]
2	Resin	Dark brown or black semi-solids It occupies 2-40% wt of crude oil Higher than asphaltene in composition Polar and non-volatile crude oil component that can dissolve in n-heptane, n-pentane, and toluene (aromatic solvent), but is not soluble in methanol and propanol. Comprises hydrogen, carbon, napthenic acid, and nitrogen.	[10]
3	Wax	High molecular weight alkanes Melting point (37 ºC < melting point < 100 ºC) Solid crystalline mixture of normal hydrocarbon (C_{20}-C_{30}) and above Plays a role in emulsion stability	[11, 12]
4	Finely divided solids	Asphaltene precipitates from bitumen. The finely sorted particles, wax crystals, sand, and clay particles stabilize emulsions. These solids prevent the thin film from flattening and contribute to the crude oil's persistence.	[13]
5	Oil-field brine	An increase in salinity results in a decrease in droplet size and an improvement in the solubilizing size of oil-water droplets. At low and high salinity, the monolayer, when restricted to a flat interface, is likely to tilt and increase tension. At moderate concentration, there is less tension due to a flat monolayer.	[14]

Types of Emulsion

Emulsion could be a dispersion of oil in a continuous water phase (oil-in-water) (O-in-W), a dispersion of water in a continuous oil phase(water-in-oil) (W-in-O), or a mixed emulsion, but water-in-oil and oil-in-water are the two major emulsions encountered in the oil and gas industry. The major emulsion types are thermodynamically unstable but can be kinetically stable for a period during their lifespan [15]. Based on kinetic stability, emulsions can be grouped into loose, tight, and medium emulsions, which differ in separation rate. For loose emulsion, separation occurs within a few minutes; for medium emulsion, separation occurs within 10 minutes; while long separation occurs for tighter emulsion.

Oil-in-Water Emulsion

For this emulsion type, the oil droplets are dispersed in the water phase. The mechanism of surfactant stabilization and adsorption within the emulsion is efficient when there is easy solubilization of the surfactant in the continuous phase. For this emulsion type, the oil phase appears as bubbles in the water, and by considering the surfactant's structure (hydrophobic and hydrophilic), the appropriate surfactant type is identified. However, in a water-oil emulsion, a water-surfactant system is more effective.

Multiple Emulsion

The shape of a multiple emulsion is more complex and comprises small bubbles suspended within larger bubbles, which are also suspended within the continuous phase. Multiple emulsions can be water-in-oil-in-water (W-in-O-in-W) and oil-in-water-in-oil (O-in-W-in-O). For W-in-O-in-W emulsion, tiny water bubbles are suspended in bigger-sized oil bubbles, which are suspended within the water (continuous phase) [16]. The type of multiple emulsion can be determined by the emulsifying agents used. The asphaltenes, waxes, resins, oil-soluble organic acids, and inorganic solids are surface-active chemicals that might be produced within the interfacial film, preventing water bubbles from coalescing. The emulsifying chemicals are fundamentally defined by hydrogen bonding with O, N-, and S- groups present in crude oil, such as Si-O and Si-OH, which also disperses in the water-oil mixture [15]. To formulate a water-in-oil emulsion, the oil phase wets the preferred chemical emulsifying agent, and the contact angle at the oil-water-solid interface is > 90 °. Despite the oil-in-water emulsion being formed, the emulsifying agent is desired to wet the water phase and achieve a contact angle < 90 °. For a contact angle closer to 90°, a stable emulsion can be formed and derived [17]. These stable emulsions are sometimes categorized as microemulsions or macroemulsions. By considering the size of the droplets, an additional type of emulsion, referred to as macro-emulsion (dispersed phase diameter > 0.1 μm), is said to be an unstable structure (thermodynamically), due to the undesirable contact between oil and water molecules, which can continuously break over time [17]. Unlike macro-emulsion, micro-emulsion yields better stability. The micro-emulsion is organically formed when two immiscible oil and water phases with ultra-low interfacial tension are combined. Micro-emulsions exhibit thermodynamic stability and have a dispersed phase diameter < 10nm. The small size of these droplets indicates that the micro-emulsion is a translucent, clear solution; also, the emulsion will break into oil phases and separate water over time [18].

Water-in-Oil Emulsion

The W-in-O emulsion behavior evaluation is of utmost importance as it exists in the exploration and exploitation of crude oil [19]. It is allocated the largest share of interest during crude production, and these emulsions need to be separated into 2 phases to meet crude oil transportation and refinery requirements [15]. Close to 95% of the crude oil globally flows to the surface as a W-in-O emulsion [20]. The W-in-O emulsion can form when crude oil mixes with aquifer water (a natural brine source), yielding water bubbles dispersed in the oil. The mixing energy needed to generate an emulsion is provided offshore by wave or wind turbulence [21]. The W-in-O emulsion is further classified into stable, meso-stable, entrained water, and unstable water-in-oil emulsions [22]. The analysis of these emulsion types is dependent on their stability. It is influenced by resin content, asphaltene content, initial oil viscosity, rheological features, and physical properties, based on more than 400 experimental analyses of oil samples and petroleum products. Of the 4 states, only meso-stable and stable W-in-O emulsions are considered emulsions. The volume of water in the W-in-O mixture is insignificant in the emulsion grouping [19]. The stable water-oil emulsion has a reddish or brownish, semi-solid appearance. The emulsion might be unchanged for a given period of time. The meso-stable W-in-O emulsion separates into free oil and water within 1-3 days, forming a brown or black fluid. The unstable W-in-O emulsion is described as crude oil with a significant water volume, and in such cases, the water remains for a limited period.

Emulsion Formation

Contact between crude oil and water, with the presence of emulsifying agents, yields crude oil emulsions. The emulsifiers and mixing volume requirements are essential during oil emulsion formation [2]. The three major criteria required for the generation of a crude oil emulsion are: two immiscible fluids being in contact, the presence of a surface-active component, and sufficient mixing to ensure dispersion of one fluid in another [22]. The velocity or pressure gradient required for emulsion production is mostly supplied by agitation, which requires sufficient energy. The right surface-active phase can be introduced to lower the energy required to yield a given bubble size. The generation of a surfactant film within the droplets yields emulsification and reduces the energy required for agitation by a factor of 10 or more. W-in-O emulsion will form when crude oil is mixed with brine and produced water droplets, which disperse within the oil, as highlighted in Fig. 1.

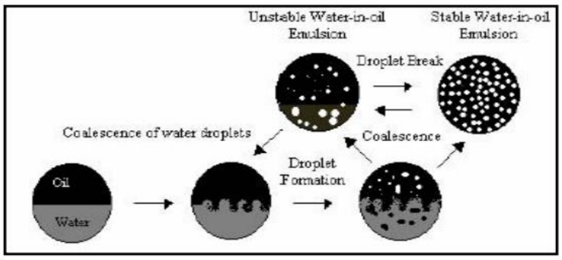

Fig. 1. Water-in-Oil Emulsion Formation [21]

Emulsion Stability

Stability is widely described as the emulsion persistence nature within a given system, and has been observed as a vital feature of W-in-O emulsions. Some emulsions rapidly return to their individual water and oil phases once removed from the surface, whereas a stable emulsion remains for a period ranging from days to years. A stable emulsion comprises a water phase, an emulsifier, and an oil phase. The emulsifier (surfactant) exists at the oil interface and speeds up the formation of a stabilized W-in-O emulsion. Emulsion stability can be considered in three scenarios: aggregation, creaming (sedimentation), and coalescence, as depicted in Fig. 2 [23].

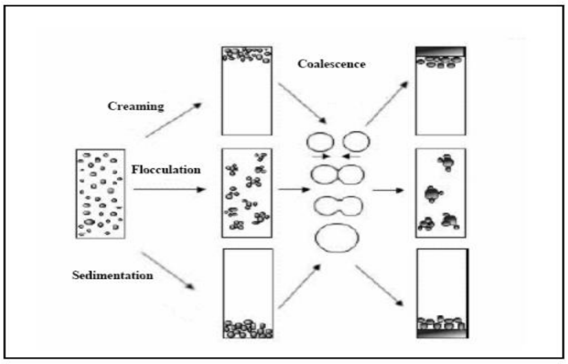

Fig. 2. Process taking place in an emulsion to yield emulsion separation and breakdown [7]

The density variation and viscosity of the two immiscible fluids are the two major factors that impact the stability of the emulsion [24].

- **Viscosity**: At high viscosities, the crude oil holds up more and larger water bubbles in comparison to low viscosities. The introduction of heat, chemicals, and diluents can significantly reduce viscosity. This, in turn, yields higher water mobility and a higher droplet settling rate, leading to coalescence and collisions and further improving the separation rate.
- **Density Difference**: The introduction of heat to the emulsion reduces the oil-density at a higher rate than the water-density, and thus yields a rapid dropout of the water. This is due to an increase in the density variation between the two phases. Removing water from light crude oil is easier than from heavy crude oil, because heavy crude oil has a density closer to that of water.

Other factors include water content, emulsion age, control of emulsifying agents, and agitation control.

- **Percentage Water**: The relative ratio between the crude oil and water in a given system impacts emulsion stability. The maximum emulsion stability is observed at low water content, so the droplets are less likely to collide with other water bubbles and coalesce. Emulsion stability decreases as the water percentage increases.
- **Age of Emulsion**: These generally increase emulsion stability. The ratio of emulsifying chemicals in the oil could increase due to photolysis, oxidation, bacterial activity, and/or evaporation of lighter hydrocarbon components. This is due to low-molecular-weight, low-density hydrocarbons like hexane, butane, and pentane present in the light ends, which can potentially vaporize a significant fraction of xylene over time. Breaking the emulsion immediately after its formation reduces the impact of aging.
- **Emulsifying Agents**: Emulsifying chemicals or Surfactants play a vital role in the emulsion production process. The surfactant can be bio-based or chemically engineered. The alteration, neutralization, or elimination of these materials prevents or resolves emulsions. The elimination of these reagents might include corrosion-inhibition procedures to reduce the amount of iron sulfide, eliminate incompatible crude oils from crude oil blends, or prevent emulsification. The alteration process includes the introduction of an asphaltene dispersant to “tie-up” the polar sites of asphaltene, the introduction of paraffin crystal enhancers to prevent emulsion stabilization by large paraffin crystals, or increasing the treatment temperature above the cloud point of the paraffin crude. The neutralization action of the emulsifying chemical is achieved by neutralizing the polar ions linked with the film of the emulsifying agent formed around the emulsified droplets. Neutralization is performed using commercial demulsifiers or coagulants that promote coalescence and accelerate gravity settling.
- **Agitation Control**: The emulsion stability could be lowered by eliminating or reducing the agitation of the oil-and-water blend. The efficiency of any demulsifier introduced into the treatment system depends directly on the extent of contact with the emulsion. Therefore, the emulsion must be thoroughly mixed after the chemical demulsifier is introduced. The rise in the mild agitation promotes coalescence. Re-emulsification could occur if the emulsion is vigorously mixed after being broken into water and oil [25].

Concept of Chemical Demulsification

Several demulsification approaches exist, including thermal, mechanical, electrical, and chemical methods. Still, chemical demulsifiers are the most widely used for emulsion mitigation and involve the introduction of reagents called demulsifiers [29]. These substances are engineered to counter the stability effect of the emulsifying reagents. Furthermore, to mitigate the impact on stability, emulsifying agents move towards the oil-water interface, disrupt or loosen the films, and promote coalescence. Optimum emulsion breaking using a chemical technique requires selecting appropriate materials for the given emulsion, using the proper chemical concentration, ensuring effective mixing of the selected reagent with the emulsion, and providing a reasonable retention time to allow water bubbles to settle in the separator. In most cases, the chemical approach is combined with the heat method to eliminate and neutralize the effects of emulsifying reagents [26].

Demulsification Mechanism

The chemical demulsification technique is a dynamic process that occurs under non-equilibrium conditions. The process aids water separation and reduces viscosity, thereby increasing the coalescence of water droplets within the emulsion. The emulsion's stability is attributed to the demulsifier, which facilitates the breakdown of the film and the separation of oil, water, and surfactant into distinct bubbles. The demulsification process is somewhat complex but can be described as a 2-step approach consisting of coalescence and flocculation (Fig. 3) [2].

- **Flocculation (Aggregation)**: This can be expressed as the first motion of demulsifiers on emulsion, and requires bringing together tiny water droplets [27]. In a flocculation process, the droplets are close to each other, form aggregates, but rarely lose their identity. Coalescence at that stage occurs only when a weak emulsifier film surrounds the water droplet. The flocculation rate can be influenced by density variations, oil viscosity, water cut, and temperature (Fig. 4).
- **Coalescence**: This is the phenomenon that deals with the rupturing of the films and the coming together of the water droplets. This is an irreversible action that is enhanced by increased flocculation rate, high IFT, high temperature, low viscosity, the absence of mechanically strong films, and water-cuts [28]. During coalescence, extra droplets join to form a single unit with a reduced perfect surface area. Coalescence can be improved by factors such as IFT, flocculation rate, chemical demulsifier, interfacial viscosity, temperature, and absence of mechanically strong films.

Another process involved in the demulsification is sedimentation. Sedimentation is the process by which a droplet of water bubbles off from an emulsion due to its superior density. In the case of the inverse process, creaming, which is the rise of oil droplets in the water, occurs. The creaming and sedimentation utilize the concept of density variation to act, and may necessarily break the emulsion. An accumulation of unresolved emulsion droplets at the oil-water interface in surface equipment forms an emulsion pad or rag layer. A pad in the surface facility presents several challenges, such as increased BS&W in the treated oil, increased residual oil in the treated water, and increased equipment upset frequency. Emulsion pads are caused by inefficient demulsifiers, low temperature, the presence of accumulating solids, and other chemicals that impair demulsifier performance.

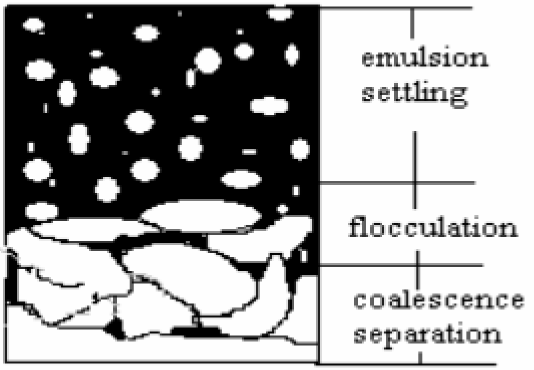

Fig. 3. The level of demulsification process of water in oil emulsion [24]

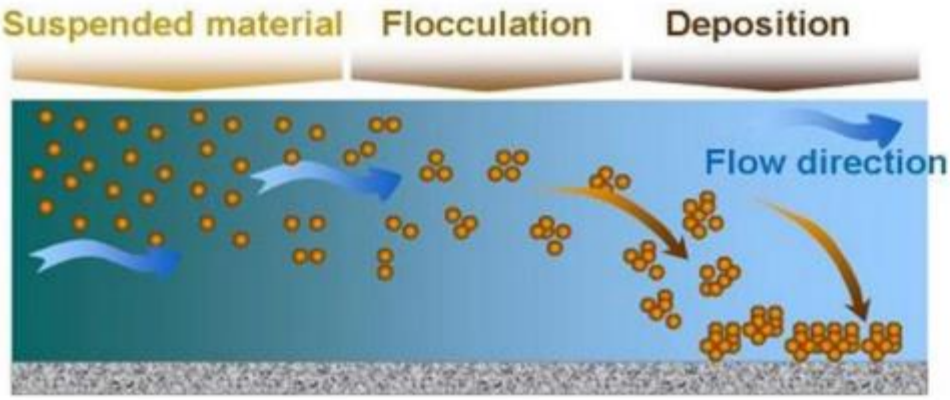

Fig. 4. Process of Flocculation [29]

Chemical Demulsifiers

Chemical demulsifiers are molecules that help to separate entrapped water from crude oil at low concentration. They prevent the formation of W-in-O emulsion. The structures of demulsifiers cannot be easily categorized, unlike those of emulsifiers. Some demulsifiers have the same structure as non-ionic emulsifiers, while others are polymers. Demulsifiers are surfactants that are vital for breaking emulsions in a given system [30]. Their molecules comprise the water-loving (hydrophilic) group and water-disliking (hydrophobic) group [31]. The hydrophilic group indicates that the substance prefers the water phase over the oil phase, whereas the hydrophobic group indicates that it prefers the oil phase over the water phase [32-34]. The best demulsifiers are those that improve destabilization and interfacial mobility of water-oil emulsions and reduce interfacial shear viscosity. Table 2 presents the key requirements for a demulsifier to achieve optimal performance.

Table 2. Feature requirement of demulsifiers for optimum quality performance [35]

S/N	Characteristics
1	Ability of the demulsifier to dissolve in the oil phase
2	The demulsifier should be able to partition between the oil and water phases.
3	The demulsifier concentration in the droplet must be enough to guarantee a high diffusion flux to the interface.
4	The demulsifier must be high enough to stop the IFT gradient, thereby hastening film drainage and promoting coalescence.

The nonionic (neutral charged hydrophilic head group), anionic (negatively charged hydrophilic head group), and cationic (positively charged hydrophilic head group) forms the three types of demulsifiers utilized to break crude oil emulsion (Fig. 5) [36].

- **Nonionic**: These are surfactants with neither positive nor negative hydrophilic head group. These chemicals are not affected by water hardness or pH, unlike anionic and cationic demulsifiers. In some cases, it is a merit that they are considered to be medium- to low-foaming chemicals. It is applicable when a very low-foaming surface is required, and examples include alkyl phenols, polyalkylene glycols, and alkyoxylated amines.

- **Anionic**: These are utilized in virtually all detergent types, which are the main component of demulsifiers. This is due to their ease of production and relatively low associated cost. The surfactant active chemicals carry a negatively charged, hydrophilic head and exhibit stable, high foaming ability. They are, however, sensitive to minerals, hard water, and pH variations. A common example is the quaternary ammonium compound, which is widely utilized in the industry. They comprise alkybenzene sulfonate (detergent), soaps (fatty acid), lauryl sulfate (foaming agent), di-alkyl sulfosuccinate (wetting agent), and lignosulfonate (dispersants).
- **Cationic**: These demulsifiers comprise a positively charged hydrophilic head group. They play a vital role as antiseptic reagents in cosmetic production as germicides and fungicides. The approach is applied when there are no cheaper alternatives. Common examples include quaternary ammonium salts, such as dimethyl dioctadecyl ammonium chloride.

Fig. 5. Basic structure of a demulsifier [36]

Requirement for Chemical Demulsifier Formulation

Different demulsifiers yield various outcomes in the demulsification process. Knowledge of the stability and formation of crude oil emulsions, and the demulsifier-type demulsification mechanism, is vital, as it can be utilized to remove entrapped water from crude oil emulsions. The parameters that are being discovered to influence demulsifier performance are:

- **Temperature**: On a laboratory scale, a 50-70 oC temperature range was considered for the demulsification process, which is similar to the dehydration process, similar to an actual refinery process. The interfacial viscosity of the internal phase will decrease with increasing temperature. This is due to the rate at which film drains, which increases with temperature. The momentum between two water droplets builds before coalescence. The two phases of the unmixed fluids are then separated due to their density differences [26].
- **pH**: The O-in-W emulsion is rarely preferred at low pH values ranging from 4-6, while W-in-O emulsions are highly preferred at high pH values ranging from 8-10. The stability of oil-water emulsion products improved as pH increased from 4-6, but further increases from 6-8 and eventually 10 yielded products with relatively little stable O-in-W emulsion and more stable W-in-O emulsion [37].
- **Droplet Size Distribution:** The droplet dimension and bubble measurement of every W-in-O or O-in-W emulsion are larger than 100 µm and 0.1 µm, respectively. The minimum droplet size distribution resulted in unrestricted emulsion

concentration and viscosity. Afterward, a longer coalescence time for the distributed drops that might either sediment (water bubble) or float (oil droplet). As the average bubble size decreases, the extended retention time reduces the rate of droplet size separation [15].

- **Oil and Water Contents**: Apart from asphaltenes and resins, water and oil contents in W-in-O emulsion are also constraints that can increase emulsion separation and stability. The presence of a demulsifier can improve the efficiency of the emulsion separation process by increasing the emulsion's water content and reducing processing time [6]. The concentration of demulsifier is vital for water separation and depends on the water content proportion (0.3) and the volumetric fraction (0.7) [15]. Emulsions with higher water content are more easily broken than those with lower water content; thus, higher water concentration yields more viscous crude oil. Furthermore, demulsification and coalescence rate continue to increase the volume of the dispersed phase; this is due to an increase in entropy, which yields efficient collisions between single-phase bubbles [38]. Contrarily, reducing the oil content from 60-90% yields little separation in a stabilized O-in-W emulsion, while reducing the oil concentration to 50% can enhance the prompt breakdown of the emulsion [2].
- **Surfactant Molecular Weight**: The molecular weight of a demulsifier enhances its mobility and diffusion features to yield adsorption kinetics at the interfacial. Demulsifiers with higher molecular weight exhibit slower adsorption kinetics, making them less active demulsifiers [39]. This might be due to the need for the demulsifier to neutralize surfactants that naturally occur in crude oil. Generally, asphaltene's adsorption kinetics in crude oil is as little as > 2-4%wt; thus, it needs a demulsifier. However, a demulsifier is required. A demulsifier with a greater molecular weight provides an adsorption interface that interacts with other particles at the interfacial film with mean adsorption kinetics [40].

Chemical Demulsification Performance Evaluation

The performance of a selected demulsifier can be evaluated at both the laboratory and field scales.

- **Laboratory Level**: At the laboratory level, selected demulsifiers are screened to determine the formulation required to achieve favorable emulsion-breaking performance. Some of the laboratory techniques include the bottle test approach, a standard method for evaluating demulsifier performance [41]. The bottle test is used to derive the water separation volume over time, the visual nature of the separated water, and the associated sludge volume. Other methods include electric dehydration test, interfacial tension measurement, rheology & viscoelasticity test, microscopy, and droplet size distribution
- **Field Level**: The field level evaluation is done to validate lab prediction under actual operating conditions. Some of the field-scale approaches include a field performance trial (pilot test), which comprises key indicators such as basic sediment & water, dehydration efficiency, desalting efficiency, quality of the separated water, chemical dosage, oil recovery, and impact analysis on the downstream [42]. Other methods include online BS&W analyzer readings, heater-treater monitoring, electrostatic treater/desalter monitoring, and water quality analysis.

Review of Chemical Demulsifier Formulation

Several demulsifiers have been used worldwide to break emulsions by various authors. Dodd summarized that demulsifiers can dissolve in both scenarios and are efficient at breaking crude oil-based emulsions, provided a small amount of sulfuric or hydrochloric acid is also added, and suggested the use of sulfuric and phenol acids [43]. De Groote postulated that the impact of a demulsifying agent, which normally comprises hydrophilic and hydrophobic groups, is that it reacts with the predominantly hydrophobic emulsifying reagent to form an adsorption complex that is hydrophilic or water-wet [44].

Salager's investigation showed that the most efficient mechanism for demulsification of a W-in-O or O-in-W emulsion is the removal of the surface-active agent from the water-oil phase by restricting the oil phase in the micro-emulsion. He also highlighted the chemical and physical phenomena involved in a dehydration process, which can be illustrated by simulating the surface-active agent naturally present in crude oil and introducing chemical demulsifiers to yield an extremely unstable emulsion [12]. Sjoblom et al. studied a real crude oil emulsion and two synthetic-based W-in-O emulsion model systems. They conducted a qualitative comparative study to explain the reactions between demulsifiers and model interfaces. They discovered that the best individual destabilizers are fatty alcohols and amines, and that the greatest destabilizing effectiveness is achieved with a fatty acid-amine blend. From their comparative study of the destabilization attributes of the modeled system and those of true crude oil, there are distinct differences and obvious similarities [45]. Staiss et al. proposed a new demulsifier comprising polyester amines. Their merit over conventional demulsifiers is greater overall movement to the interfaces, enhanced emulsion coalescence and breakage, enhanced corrosion inhibition, and improved water quality [46]. According to Aamir's study, the blend of sulfonic acid and phenol showed the best performance as a chemical reagent for separating trapped water from crude oil [47]. Al-Shamrani et al. utilized a solubilized air flotation approach to separate crude oil from polluted water. They report that flocculation and coagulation are vital pre-treatment methods for solubilized air flotation to enhance oil recovery in the process. The utilization of inorganic forms of certain coagulants, such as aluminum sulfate, improved the separation technique [48]. Hanapi's investigation using a blend of water-soluble and oil-soluble demulsifiers yielded excellent water separation. He also observed that the designed demulsifiers were better than other commercial demulsifiers [49]. Nikkhah et al. developed a nanotitania-based chemical demulsifier for the W-in-O emulsion. In their evaluation, a sol–gel approach was used as a substitute to design the powder-titania particles. Then, the evaluation continues with transmission electron microscopy (TEM), particle size analysis (PSA), X-ray diffraction (XRD), electrostatic testing, standard IP77, and bottle test approaches. From their results, nanotechnology achieved up to 90% efficiency in water separation and reduced the separation time [50]. Conducted studies have shown that ionic solutions can be utilized to separate W-in-O emulsion [51]. In the experimental investigation conducted by Hazrati et al., it was proposed that liquids with long alkyl chains, blended with PF6, NTf2, and Cl anions, serve as a functional demulsifier [52]. The experiment was conducted by measuring interfacial tension (IFT) between crude oil and water at 1-, 2-, 6-, and 24-h time points, followed by a bottle-test analysis. The alkyl chain length, anion type, and IL dosage were varied at three levels to achieve good demulsifier behavior. As a result, they achieve water-separation effectiveness of 86–95% and an IFT value of 0.7–6.26 mN/m. They summarized that higher demulsification efficiency is obtained at longer alkyl chain lengths for ionic liquids, corresponding to higher IFT. Feitosa et al. investigated cardanol-based additives to evaluate their demulsification mechanisms [53]. The three Brazilian crude oils with 60-240 g/L NaCl of salinity, 30% (v/v) brine cut, at a pH value of 3-10, in a constant mixing rate (3200 rpm) were utilized. Four types of chemical reagents (hydrogenation, formaldehyde polycondensation, ethoxylation, and ethoxylation of formaldehyde polycondensation) were used to design end products from cardanol. 1H NMR and FTIR studies were done to comprehensively describe the reagent. Using the bottle test approach, the experimental study was conducted at 60 °C, and an additional 200 ppm of the chemical was tested. Their results indicate that at neutral pH, ethoxylated compounds are more suitable for separating W-in-O emulsions. Due to the economic and ecological impacts of conventional demulsifiers on developing countries, several authors have suggested local alternatives.

Udonne carried out a demulsification of crude oil using sulfuric acid and castor oil. From the result of his experimental study at 500rpm and 1000rpm, sulphuric recovered 1.25% and 2.5% of entrapped water, respectively, while castor oil recovered 2.5% and 6.25%, respectively [54]. Emuchay et al. formulated 3 sets of local demulsifiers (comprising liquid soap, starch, camphor, calcium hydroxide, paraffin wax, coconut oil, and d-limonene), namely blends A, D, and E. They compared them with two conventional demulsifiers (EC-2606A and EC-2303A). From the results of their experimental study, the local demulsifiers yield the best performance, with separation above 80% water [55]. Francis et al. conducted a comparative demulsification study between a conventional demulsifier (separol) and a locally designed demulsifier (a mixture of camphor, jatropha oil, paraffin wax, cassava starch, and liquid soap) over varying temperature ranges. From their experimental study, the locally designed demulsifier yielded a better performance at all temperature variations as it recovered 2-10 ml of water [56]. Amajuonyi et al. conducted an experimental study on the formulation of demulsifying agents for the treatment of water-in-oil emulsions. The study evaluated the effectiveness of two demulsifiers, a locally formulated demulsifier (LFD) and a commercially available imported demulsifier (H_2SO4), in treating water in crude oil emulsion. The material, including alum, carter oil, starch, liquid soap, camphor, and a sample of crude oil emulsion used in the study, was obtained from an oil field in the Niger Delta region of Nigeria. The centrifugal agitation method was used to determine the most effective demulsifier in breaking the emulsion. The results showed that LFD was more effective at breaking down the emulsion than H_2SO_4. The study concludes that locally formulated demulsifiers can be a cost-effective and efficient alternative to commercial demulsifiers in the oil industry [57]. Falode and Aduroja compared the demulsification performance of a commercial demulsifier with locally prepared demulsifiers from calotropis procera, jatropha curcas, thevetia perrieri, terminalia catappa, carica papaya, and citrus limon. From results obtained using a combination of bottle tests and response surface methodology, their formulated demulsifier performed favorably compared with the commercial demulsifier [58]. Victor-Oji et al. compared the demulsification performance of cashew nut shell liquid and a commercial demulsifier. Based on their study, cashew nut shell liquid performed favorably with the commercial demulsifier and could potentially replace it when the economics are favorable [59]. Mepaiyeda et al. compared the demulsification performance of a commercial demulsifier with a local ionic demulsifier derived from mixing plant extract, coconut oil, starch, and liquid soap. Based on the results of their experimental study, the formulated demulsifiers demonstrated better demulsification performance than the commercially available demulsifiers [60]. Olanrewaju, et al. Carried out an experimental study on the development of a bio-based demulsifier for water-in-oil emulsion treatment using plant extracts from Jatropha curcas seeds. Their study developed a bio-based demulsifier for treating water-in-oil emulsions using plant extracts from Jatropha curcas seeds. The plant extracts were obtained by solvent extraction and formulated by dissolving them in a mixture of ethanol and water. The formulated demulsifier was tested on a W/O emulsion prepared from crude oil and water. The results showed that the formulated demulsifier was effective in demulsifying the W/O emulsion, with a demulsification efficiency of up to 75% [61]. Azubuike and Eiroboyi conducted a comparative demulsification study of a commercial demulsifier, sodium dodecyl sulfate (SDS), and a locally formulated demulsifier derived from Nicotiana tabacum stalk ash and leaf extract. Crude oil emulsions from two oilfields were used for evaluation. Based on the results of their experimental study for the first oilfield, the commercial demulsifier performed best, achieving 73.33% water-separation efficiency. In comparison, SDS and the locally formulated demulsifier recorded 61.67% and 66.67% water separation efficiency, respectively. In their experimental study of the second oilfield, the commercial demulsifier yielded the best performance, achieving 86.67% water separation efficiency. In comparison, SDS and the locally formulated demulsifier achieved 65.83% and 78.33% water-separation efficiency, respectively [62]. Allen and Amiebibama evaluated demulsification performance using orange peel oil (OPO), soya bean husk oil (SBHO), and a commercial demulsifier phase treat (PT). From the results of their experimental study at 1000 ppm concentration, OPO yielded 60%, SBHO yielded 45%, and PT recorded 87.5% crude oil separation, respectively [63]. Ndubuisi et al. compared the demulsification performance of a locally formulated demulsifier (camphor, olive oil, shea butter, starch, distilled water, and liquid soap) with that of a conventional demulsifier. From their experimental study, the locally formulated demulsifier performed better than the conventional demulsifier in a low-temperature-low-time system, as it recovered 4-5.5 ml of entrapped water. The demulsification performance of the locally formulated demulsifier remained fairly constant up to 60 °C [64]. Okereke et al. conducted a comparative demulsification study of locally formulated demulsifiers 1 (CONK1) and 2 (CONK2). In their experimental study, CONK1 performed better than CONK2, recovering 1-7.5 ml of water, while CONK2 recovered 2.5-5 ml of water [65]. Okafor et al. conducted a demulsification performance study using an agro-based demulsifier from corn cobs from furnace burner ash (FBA) and microwave ash (MWA), and a chemical-based demulsifier [66]. Based on the study's results, the MWA demulsifier yielded the best demulsification performance, with 72% separation efficiency, while the chemical demulsifier and FBA recorded 60% and 39%, respectively. The methodology utilized by previous authors has been summarized in Table 3.

Table 3. Methodology utilized by previous authors

S/N	Ref.	Demulsifier	Content	Conc	Temp. conditions	Speed conditions
1	[54]	Sulphuric Acid	Conventional	1-6 drops	-	500, 1000, and 1500 RPM
		Castor Oil	Castor oil	1-6 drops	-	
2	[55]	Blend-A	Starch, distilled water, limonene, coconut, calcium hydroxide, and liquid soap	0.2ml 0.4ml 0.6ml 0.8ml 1.0ml	-	-
		Blend-D	Camphor, liquid soap, distilled water, starch, and coconut oil,			
		Blend-E	Camphor, coconut oil, liquid soap, distilled water, starch, petroleum wax, and calcium hydroxide			
		EC-2303A	Conventional			
		EC-2606A	Conventional			
3	[56]	Local demulsifier	Jatropha oil, camphor powder, paraffin wax, starch, liquid soap, and distilled water	1ml	27 °C 35.6 °C 48.2 °C 48.8 °C	-
		Separol	Conventional			
4	[57]	Local demulsifier	Alum, castor oil, starch, liquid soap, camphor, and distilled water	0.2ml, 0.4ml, 0.6ml, 0.8ml, 1ml, 1.2ml	85 °F	1000, and 1500 RPM
		Sulphuric acid	Conventional			
5	[58]	Local demulsifier	Calotropis procera, jathropha curcas, thevetia ferifolia, terminalia catappa, carica papaya and citrus limonium	4g/ml	70°C	-

		W054 chemical demulsifier	Conventional			
6	[59]	Cashew nut shell extracts	Fluid from the cashew nut shell	10ppm, 20ppm, 30ppm, 40ppm, 50ppm	60°C	-
		Phase treat (PT) 4633	Conventional			
7	[60]	Liquid formulated demulsifier	Plant extract, coconut oil, starch and liquid soap	0.034ml 0.102ml	27°C	
		Commercial demulsifiers	Conventional		27°C	
		Locally formulated demulsifier	Nicotiana tabacum leaves, extracts, seed oil, and stalk ash		27°C	
8	[61]	EO/PO block copolymer	Conventional	100ppm 200ppm 300ppm	27°C	
		Sodium dodecyl sulphate	Conventional		27°C	
		Orange peel oil	Extract from orange peel			
9	[63]	Soya bean husk oil	Extract from soya bean husk	0ppm 200ppm 400ppm 600ppm 800ppm	-	500, 1000 and 1500 RPM
		Phase treat	Conventional			
10	[64]	Locally formulated demulsifiers	Camphor powder, coconut oil, paraffin wax, cassava starch, liquid soap	0.2ml 1ml	20°C, 40°C, 60°C	-
		Imported demulsifier				
11	[65]	Locally formulated demulsifier-1	Alum, castor oil, starch, liquid soap, and camphor	0.2ml 0.4ml 0.6ml 0.8ml 1.0ml 1.2ml	60°F	-
		Locally formulated demulsifier-2	Alum, castor, starch, liquid soap, camphor, and xylene			
		Furnace Burner Ash-based Corn-Cob	Blend of Corn-cobs and Ethanol			
12	[66]	Microwave Ash-based Corn-Cob	Blend of Corn-cobs and Ethanol	5ml	-	-
		Sulphuric Acid	Conventional			

Challenges Associated with Chemical Demulsification Using Local Materials

Demulsifiers have recorded impressive performance, but the oil and gas industry is continually faced with new challenges. These challenges are grouped into subsurface and surface-based challenges. The sub-surface includes high water-cuts in mature reservoirs, and wells produced by tertiary/enhanced oil recovery, while the surface includes the demulsifier and operational-based challenges [67, 68]. The operational challenges include the heat required and offshore operating conditions, while demulsifier challenges include sludge formation, biodegradation, and emulsion quality [69, 70].

- **Sludge Formation**: Some of the locally formulated demulsifiers comprise additives that do not blend homogeneously but form a colloidal phase when stirred together, only to return to separate phases when not stirred. When these demulsifiers are introduced into the emulsion, some particles and sediments from the demulsifiers drop to the bottom to form sludge (water-in-oil) comprising water, hydrocarbons, and sediments [71].
- **Biodegradation**: The locally formulated demulsifiers are most biodegradable and decay by the action of bacteria and microorganisms. This is a critical parameter that significantly reduces their ability to compete effectively with conventional materials [72].
- **Crude Oil Quality**: The quality of the crude oil poses an obstacle to the performance of the demulsifiers. The quality of crude oil is tied to the recovery method used to produce it. In enhanced oil recovery, associated reagents such as polymers, surfactants, and alkalis aid recovery, alter its physicochemical properties, and increase its asphaltene & resin content. This ultimately leads to the formation of a complex stable multiple emulsion [73].
- **Nature of the Separated Water**: The nature and quality of the separated water can significantly impact the choice of demulsifiers to be utilized. The increase in salinity of the separated water negatively impacts the efficiency of the demulsifiers [74]. In the event of separation, the resulting separated water poses an ecological challenge due to its dissolved salts.

Probable Solutions to the Challenges

Given the challenges associated with the locally formulated demulsifier, the following research paths are recommended.

- Comprehensive study of physicochemical properties, asphaltene, and resin content properties of the crude oil to be studied.
- Comprehensive study of demulsification mechanism for each recovered crude oil to provide a theoretical base for the formulation and synthesis of demulsifiers.
- The formulation of dual-function demulsifiers suitable for breaking O-in-W emulsion, W-in-O emulsion, and complex emulsion.
- Formulation of demulsifiers with high molecular weight for easy formation of micelles suitable for solubilizing emulsifying agents to break emulsions.
- Improve the aromacity and the wettability of the formulated demulsifiers. The aromacity of demulsifier is enhanced by modifying it with aromatic compounds and by improving the initiator's aromacity.
- Formulate demulsifiers with more soluble particles and fewer colloidal additives, and the proper operational speed and temperature.
- Utilization of chemicals that will be inert to the demulsification performance of the demulsifiers, but can preserve the demulsifier.
- Simultaneous utilization of chemical demulsification with desalination process [75].
- Optimization of demulsifier and desalination performance using algorithms such as response surface methodology [76].

Conclusion

This study presents a review of demulsification and the use of locally formulated demulsifiers in the Niger Delta. The concepts of emulsions, demulsification, solutions, and locally formulated materials were explored and evaluated.

- A comprehensive understanding of the emulsion formation concept, mechanism, and type is necessary for the design of a suitable demulsifier
- Nicotiana Tabacum, corn cobs, orange peels, soybean husk, and moringa seeds showed promising signs for crude oil demulsification. Camphor powder is a key ingredient in the formulation of crude oil demulsifier. Alum facilitates sediment settling, while liquid soap serves as a binder, allowing the demulsifier to link the lipophilic and hydrophilic ends.
- Formulation of demulsifiers with soluble additives and fewer colloidal particles is required to reduce sludge formation
- Speed and Temperature increase the demulsification of locally formulated demulsifiers
- The demulsification performance of the local demulsifier can be further optimized using software such as response surface methodology.

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