



Micro-emulsion-based dissolver for removal of mixed scale deposition

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Abstract

The term mixed scale pertains to the scales found in oil and gas production system containing both organic and inorganic constituents in such a way that either aqueous-based inorganic dissolver or solvent-based organic dissolver fails to act on it. These scales are also known as wetted scales. This research discovers formulations which can effectively dissolve and disperse mixed scales dominated by inorganic content. Micro-emulsion-based solutions are identified as the best in tackling such mixed scales. A few inorganic and organic dissolving chemicals along with surfactants and co-surfactants are considered in this research to develop environment friendly solutions. The stable micro-emulsions are subjected to detailed dissolution study to establish their efficacy. The synthesized chemical solutions are shown to dissolve mixed scales of different composition. A chelant-based micro-emulsion formulation is also found to be effective in dissolving difficult to treat metal naphthenate scales co-precipitated with organic content, which is a novel application.

Keywords Mixed scale · Inorganic · Organic · Micro-emulsion · Surfactant · Co-surfactant

Abbreviations

ME	Micro-emulsion
MAPE	Mono alkyl palm ester
HCl	Hydrochloric acid
DLABSA	Derivative of linear alkylbenzene sulfonic acid
GE	Glycol ether
ASA	Alkyl sulfonic acid
EDTA	Ethylenediamine tetraacetic acid
DTPA	Diethylene triamine pentacetic acid
HEDTA	Hydroxyethyl ethylene diamine triacetic acid
CHEL	Chelant
PA	Primary alcohol
ARN	Aromatic naphtha
XY	Xylene

Introduction

Scale formation, both organic and inorganic, is recognized as one of the important operational problems in oil and gas production equipment and facilities both surface and

subsurface (He et al. 1996; Sousa et al. 2019; Tavakkoli et al. 2013). It is also a predominant cause of formation damage problem in the mature oil-producing regions of the world (Sharma 2006). Production impediments caused by organic and inorganic deposits including naphthenate in the wellbore and flow lines are also a major flow assurance challenge (Jamaluddin and Kabir 2012; Tavakkoli et al. 2014). In deep-water environments, these production issues are severe owing to cooler temperatures, greater hydrostatic head and longer subsea tiebacks.

Organic and inorganic scales are formed in production system because of reduction in the solubility of precipitating species affected by changes in temperature, pressure, composition and pH. Scales can be inorganic like calcium carbonate, calcium sulfate, barium sulfate and strontium sulfate or organic like paraffin and asphaltene. In practice, the occurrence of mixed scales is common and creates a challenging treatment and remediation problem (Al-Taq et al. 2015). Mixed scales dominated by inorganic content are formed by predominantly inorganic species co-precipitating with organic material. The resulting highly complex structured scales are difficult to treat and require aggressive, severe and costly remediation techniques (Kelland 2009). In the scale matrix, the organic species can be present in such a way that it forms a protective coating on inorganic particles, thereby rendering them resistant to acid attack.

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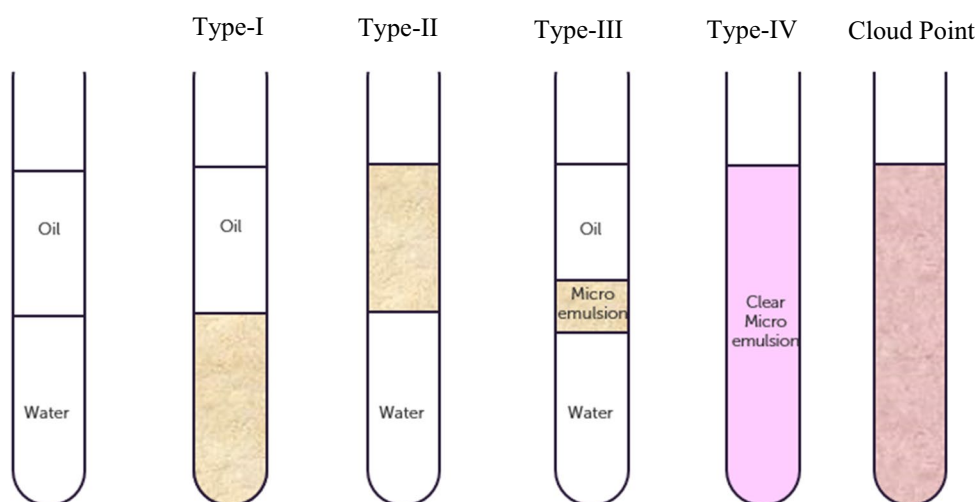
The ideal way to chemically treat such mixed scales will be to have a mixture of an inorganic dissolver (acid or chelate) and an organic dissolver (organic solvent). But due to the immiscibility of these two components, their stable mixture alone cannot be formed. These two components may form emulsion (macro-emulsion) with the help of surface active compounds. But emulsions are not in equilibrium and emulsion-based formulations cannot be used. Micro-emulsion presents another option for achieving stable mixture of these two immiscible components (Israelachvili 1994). Micro-emulsion is thermodynamically stable mixture of a polar fluid phase (e.g., water) and a non-polar fluid phase (e.g., organic solvent) brought about by a surfactant and a co-surfactant (commonly a short chain alcohol). In a micro-emulsion, the droplet size is usually less than 100 nm.

Interest in micro-emulsion dates back to nineteenth century when several such formulations were used advantageously in leather, machine tool and laundry industries. The present understanding in micro-emulsion owes much to the pioneering work done in the middle of twentieth century (Hoar and Schulman 1943; Schulman et al. 1959). Winsor described the micro-emulsion formation phenomenon in the form of various types of stages presented in Fig. 1 (1948). Initially the oil and water phases are separate. Type-I (O/W) and Type-II (W/O) emulsion can be formed by the addition of a surfactant/co-surfactant system. In Winsor Type-I, the surfactant-rich water phase accompanies with the oil phase and the surfactant exists as monomer at small concentration. In Winsor Type-II, the surfactant-loaded oil phase combines with the surfactant-poor aqueous phase. If the surfactant system is capable of forming a micro-emulsion, a third phase, Winsor Type-III will appear at the interface of oil and water. In this microemulsion, both the water and oil are

surfactant-deficient phases. Type-III typifies attainment of reaching ultra-low interfacial tension and is used profusely in enhanced oil recovery studies for screening of surfactants (Hirasaki et al. 2011; Bera and Mandal 2015). The same surfactant system if added more will convert the whole liquid into micro-emulsion, which is classified as Type-IV. A Winsor Type-IV microemulsion is an extension of a Winsor Type-III at higher surfactant concentrations, where the middle phase extends and becomes a single phase. Further addition of the surfactant system will cause phase separation which is described as cloud point.

The petroleum industry is conscious of the micro-emulsion technology and is used as an efficient tool in enhanced oil recovery techniques (Sharma and Shah 1985). In the oil and gas drilling, the micro-emulsion concept has been applied for filter cake removal and wellbore displacement (Brege et al. 2012). The surface application involves cleaning of separators for removing oily material (Quintero and Carnahan 2013). Using mesophase formulation, there is only a recent attempt for the cleanup of wellbore deposits concentrated with paraffin and asphaltene (Quintero et al. 2017). The novelty of this research is the development of micro-emulsion-based solutions targeting dissolution of oil field deposits which are mixed in nature and dominated by inorganic content. The successful dissolution tests are discussed in “Results and discussion”. The treatment of metal naphthenate mixed scale is also explained using the newly formulated micro-emulsion concoction, thereby providing a new tool to the Oil and Gas Production Technology (Misra et al. 2018). Another uniqueness of this work is in choosing environment friendly chemicals with respect to the nature. The micro-emulsion formulations developed are detailed in “Chemical formulations”.

Fig. 1 Winsor classification of emulsions (Winsor 1948)



Chemical formulations

In a micro-emulsion (ME), both the aqueous-based and the oil-based phases coexist in an isotropic and thermodynamically stable state as a single-phase fluid (Danielsson and Lindman 1981). Such emulsions are transparent to translucent. Inorganic dissolver such as acid or chelate and organic dissolver such as solvent are the main active ingredients in the proposed formulations. Surfactant and co-surfactant chemicals are also required to allow the mixture of the main ingredients to become micro-emulsion (Solans and García-Celma 1997). Not all the surfactants/co-surfactant systems can create micro-emulsion, and not all stable formulation can be useful to the current problem of dissolving the mixed scale. The phase study and experience from trialing many inorganic and organic dissolving chemicals along with different surfactant and co-surfactant combination will be presented as another article. Only the stable micro-emulsions which can dissolve the mixed scales and do not create sludge as reaction products are discussed here.

As an endeavor to develop environment friendly formulations, mono alkyl palm ester (MAPE) is tried as a solvent. The MAPE is an ester of fatty acids obtained from palm oil. Mono alkyl palm ester is a biodiesel and is used as a fraction of commercial diesel (May et al. 2005). The first solution of micro-emulsion thus prepared is MAPE–HCl–DLABSA–GE system, where a derivative of linear alkylbenzene sulfonic acid (DLABSA) is used as the surfactant and glycol ether (GE) is used as the co-surfactant, respectively, together with hydrochloric acid (HCl). The glycol ether selected is inexpensive of very low volatility and toxicity. Even when using the biodegradable LABSA derivative, there is still room for improvement of our environment because benzene and alkyls originate from distilling oil and are thus non-sustainable chemicals.

Among the acid dissolvers besides hydrochloric acid, alkyl sulfonic acid (ASA) is attempted for scale treatment. It is used in catalysis of many organic reactions and for rust cleaning products because unlike sulfuric acid it does not cause acid attack (Laffitte and Monguillon 2013). Being a weaker acid than hydrochloric or sulfuric acid, alkyl sulfonic acid can be used with advantage where slow reactions are desired. The other advantage is that alkyl sulfonic acid is easily biodegraded (Toxnet Database 2018). It is, therefore, used to make stable micro-emulsion with mono alkyl palm ester as solvent. The second solution of micro-emulsion so prepared is MAPE–ASA–DLABSA–GE system.

Similarly, chelants of amine-acetic acid class like ethylenediamine tetraacetic acid (EDTA), diethylene triamine pentacetic acid (DTPA), hydroxyethyl ethylene diamine

triacetic acid (HEDTA) which are gaining attention because of its biodegradability and acid solubility can be applied as acid dissolvers (Theptat et al. 2013). If acidic solution of chelant (CHEL) can be made part of a micro-emulsion with solvents, it can be a good proposition for the current research. Encouraging results are obtained using a derivative of LABSA and a primary alcohol (PA) as surfactant and co-surfactant, respectively, and with aromatic naphtha (ARN) as the solvent. Thus, the third micro-emulsion solution is ARN–CHEL–DLABSA–PA system. The primary alcohol chosen is largely non-toxic and can be rapidly degenerated by photo-degradation.

The micro-emulsion formulation having composition at any point within the stability envelope will be effective. The final choice of the component concentration is dictated by the nature of scale and cost effectiveness in terms of the surfactant. For example, if organic species is more in the deposit, the choice will shift towards higher concentration of solvent.

Results and discussion

Many of the scales formed during petroleum production are mixed scales where inorganic species like calcium carbonate or barium sulfate gets deposited along with organic species like wax or asphaltene (Ramones et al. 2015; Newberry et al. 2017). For the purpose of determining dissolution capacity of the prepared chemical formulations, mixed scale deposit samples are obtained from two different fields of varying fluid properties. Table 1 shows the composition of the mixed scale. Sample 1 is used for qualitative assessment and Sample 2 is used for quantitative assessment of the chemical formulations. These are good examples of mixed scale dominated by inorganic content where neither acid nor aromatic solvent can dissolve the deposit as observed in Fig. 2. In the dissolution test with hydrochloric acid (50% concentrated) and xylene, 2 g of mixed scale Sample 1 is taken in a test

Table 1 Composition of the mixed scale

Component	Sample 1 (weight %)	Sample 2 (weight %)
Wax	8.2	28.7
Asphaltene	0.5	1.75
Naphthenates	0.3	1.05
Crude oil	0.7	2.45
Water	0.3	1.05
Limestone	83.7	60.45
Ferric oxide	2.7	1.95
Silica	1.8	1.3
Clay	1.8	1.3

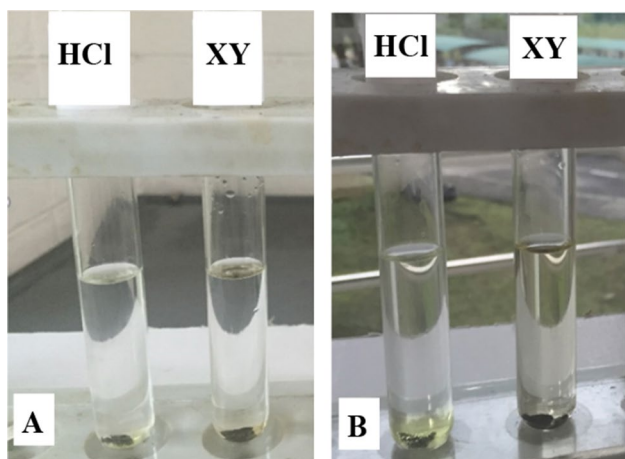


Fig. 2 Mixed scale deposit Sample 1 in different solutions of hydrochloric acid (50% concentrated) and xylene. **a** Picture captured immediately after the deposit and solution come in contact. **b** Picture captured after 2 h of dissolution

tube and 10 ml of the solution is added to it. The test is performed at room temperature. The visual inspection conducted shows no substantial dissolution in hydrochloric acid or xylene (XY) even after 2 h of observation. The micro-emulsion formulations created in this research can very well tackle these mixed scales.

Dissolution test with mixed scale

For dissolution check, three test tubes are taken into which 2 g of Sample 1 is added to each test tube. 10 ml of different chemical formulation prepared in this research is added to each of the test tubes. The analysis is performed at room temperature. The visual examination shows good dissolution

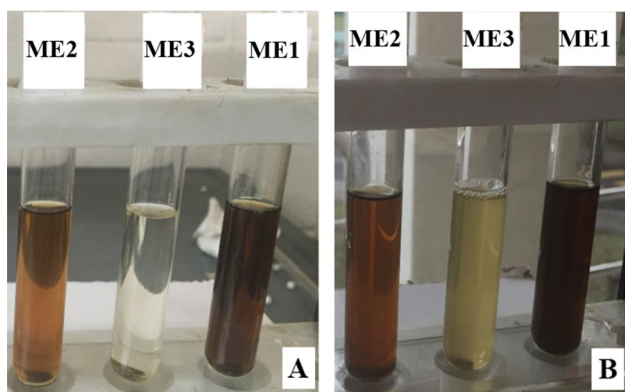


Fig. 3 Mixed scale deposit Sample 1 in different solutions of micro-emulsion formulation (ME1: MAPE–HCl–DLABSA–GE system; ME2: MAPE–ASA–DLABSA–GE system; ME3: ARN–CHEL–DLABSA–PA system). **a** Picture captured immediately after the deposit and solution come in contact. **b** Picture captured after 2 h of dissolution

in all the three micro-emulsion formulations. Figure 3 displays the images of the visual inspection for dissolution assessment of the micro-emulsion formulations. Comparing the dissolution screening from Figs. 2 and 3, it is concluded that the prepared micro-emulsion formulations are better at dissolving the mixed scale in comparison with hydrochloric acid and xylene.

For further detailed analysis on the dissolution capacity of the micro-emulsion formulation, dissolution tests are performed on Sample 2 at 40, 50 and 90 °C. Before the dissolution tests, it is ensured that the developed micro-emulsion formulations are stable at elevated temperatures commonly encountered in oil fields. In the detailed dissolution study, 2 g each of the deposit Sample 2 is taken in five test tubes labeled for 1, 2, 4, 8 and 24 h. 10 ml of a micro-emulsion formulation is added to each of the five test tubes. The tubes are placed in water bath at the desired temperatures. At the intervals of 1, 2, 4, 8 and 24 h, the corresponding test tube is removed and the contents are filtered under vacuum. The contents are then washed twice with 5 ml of water and the filtrate is dried in an oven. The dried contents are ignited in the Muffle furnace at 550 °C. The residue after decomposition is digested in 10 ml of 1 M hydrochloric acid and filtered. The filtrate is made up to 100 ml in a volumetric flask. Calcium content in such solutions is measured and converted to calcium ppm equivalent to 10 ml formulation. In this way, the dissolution in terms of calcium content is measured, because it is the dominating species in the deposit sample. The results of dissolution test are provided in Figs. 4, 5 and 6. The error in experimental measurement can be up to 4%.

Figure 4 shows the outcome of dissolution tests performed on Sample 2 with the formulation of MAPE–HCl–DLABSA–GE system. It is noticed that the reaction is faster with increase in temperature from 40 to 90 °C. The observed maximum of calcium dissolution at 40, 50 and 90 °C is 24,170, 25,960 and 27,000 ppm, respectively. As Sample 2 has 60.45% lime stone, this then

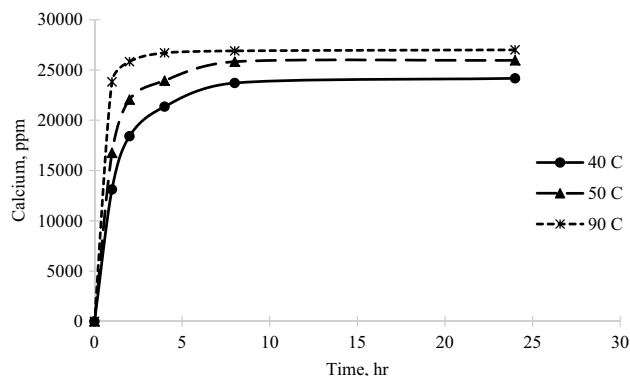


Fig. 4 Dissolution study of mixed scale Sample 2 with MAPE–HCl–DLABSA–GE system

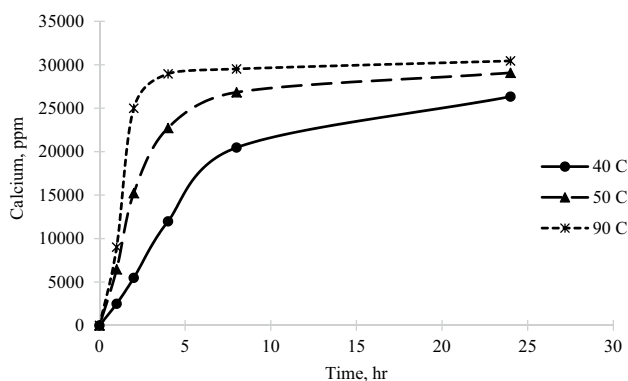


Fig. 5 Dissolution study of mixed scale Sample 2 with MAPE–ASA–DLABSA–GE system

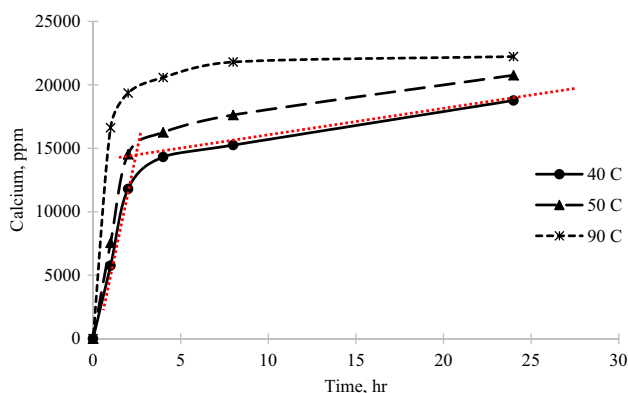


Fig. 6 Dissolution study of mixed scale Sample 2 with ARN–CHEL–DLABSA–PA system

translates to 40.0, 42.9 and 44.7 g/lit maximum deposit dissolution capacity, respectively.

Figure 5 shows the results of dissolution tests performed on Sample 2 with the formulation of MAPE–ASA–DLABSA–GE system. It is observed that at 40 °C the reaction rate in the initial time scale of up to 2 h is less compared to the MAPE–HCl–DLABSA–GE system from Fig. 4. This is because alkyl sulfonic acid is a weak acid. But with an increase in the temperature sufficiently fast reaction rates are detected. The observed maximum of calcium dissolution at 40, 50 and 90 °C is 26,340, 29,080 and 30,450 ppm, respectively. This then translates to 43.6, 48.1 and 50.4 g/lit maximum deposit dissolution capacity, respectively. The maximum dissolution obtained with MAPE–ASA–DLABSA–GE system is more in comparison with MAPE–HCl–DLABSA–GE system.

Figure 6 shows the outcome of dissolution tests performed on Sample 2 with the formulation of ARN–CHEL–DLABSA–PA system. The chelant uniquely has multiple coordination sites and is soluble even in low pH. This ability makes it a good dissolver for calcite. From

Fig. 6, it can be identified that at higher temperature a better kinetics is observed. The observed maximum of calcium dissolution at 40, 50 and 90 °C is 18,770, 20,760 and 22,230 ppm, respectively. This then translates to 31.0, 34.3 and 36.8 g/lit maximum deposit dissolution capacity, respectively.

Another information from Fig. 6 is that the dissolution takes place in two stages based on the mapped dotted red line. The first phase is the fast acid attack during the initial time scale. The second stage is slow and is the chelating reaction. Because of good chelation characteristics, this system also responded well to metal naphthenate dissolution which is discussed in “Dissolution test with metal naphthenate co-precipitated with organic material”. At 90 °C, the curve in Fig. 6 shows that the chelant is acting more like an acid.

A major factor governing the dissolution of mixed scale by all the three micro-emulsions is the choice of inorganic dissolver (acid or chelate), because these mixed deposits are dominated by inorganic content. Acids are known to react stronger than chelating agents with calcite. Common to all the micro-emulsions discussed here, an increase in temperature enhances the removal of mixed scale. This can be explained using the Arrhenius equation. Increasing the temperature increases the number of high energy collisions which possess the activation energy for the reaction between solution and deposit, leading to more dissolution of the deposit at higher temperature.

Dissolution test with metal naphthenate co-precipitated with organic material

Naphthenate mixed scale issues are not new, but the potential severity and increased occurrence highlight the need for successful prevention and remediation management (Mohamed et al. 2016). A metal naphthenate deposit obtained from field has 60% organic content by ignition. From the collected ash, upon analyzing for cations the composition of di/trivalent cations is presented in Table 2. It is observed that iron dominated other cations, and is followed by chromium.

Table 2 Composition of di/trivalent cations in the metal naphthenate ash

Cation	Concentration (wt%)
Barium	0.42
Calcium	7.12
Chromium	12.61
Manganese	0.86
Strontium	0.03
Zinc	0.24
Iron	78.69

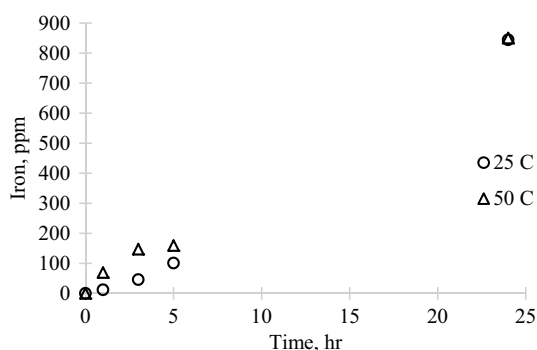


Fig. 7 Concentration of dissolved iron over time in the metal naphthenate dissolution test at 25 °C and 50 °C

This indicates that the metal naphthenate deposit is predominantly originated from corrosion products.

This metal naphthenate deposit is subjected to dissolution with the chelant-based micro-emulsion formulation (ARN–CHEL–DLABSA–PA system) at 25 and 50 °C. Figure 7 shows iron concentration obtained over time in the dissolution which is an indicative of the reaction propagation. It is observed that increase in temperature has an impact only during the initial time scale. This may be because of the attainment of solubility limit at longer time.

It is known that metal naphthenate can be hydrolyzed by dilute hydrochloric acid in the presence of toluene under reflux (Mohammed and Sorbie 2009). The progression of this reaction is possible because as soon as metal naphthenate is hydrolyzed, the naphthenic acid partitions with toluene. The metal ion is partitioned in the hydrochloric acid. Micro-emulsion is providing the same opportunity because both dispersed and continuous phases work in tandem. Figures 6 and 7 show that besides mixed scale removal, chelate-based micro-emulsion formulation also provides good activity against metal naphthenate mixed scale.

Conclusion

This research considers the development of environment friendly formulations which can remove mixed scales dominated by inorganic content. Along with depositing in production system, such mixed scales are also reported at formation faces causing formation damage and losing well productivity. In mixed scale, the organic matter is so entrapped in the scale matrix that it comes in the way of scale dissolution. Normally, hydrochloric acid which is good enough to dissolve calcite scale failed to attack the mixed scale even in concentrated form of the acid. In this scope, three stable micro-emulsion formulations are developed with organic solvents and calcite dissolving chemicals with the help of surfactants and co-surfactants.

Emphasis is given on selection of chemicals which are environment friendly. The developed micro-emulsion-based formulations allow dissolution and dispersion of organic matter so that acid/chelant can make contact with the exposed inorganic surface. The dissolution tests performed with the prepared micro-emulsion solutions on the mixed scale deposits from different fields show impressive results. Naphthenate mixed scale, which is even tougher to handle than just the naphthenate scale, can also be dissolved using the chelant-based micro-emulsion formulation prepared in this work. The results obtained in this research are very promising and will lead to formulating more and better micro-emulsions in future for mixed scale problem.

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