

Effect of EDTA Addition on Acidizing Treatment Process

Pengaruh Penambahan EDTA pada Proses Acidizing Treatment

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Abstract

Acidizing treatment is commonly used to solve scale problem on production equipment. In this process, Hydrochloric acid (HCl) is often used to treat CaCO₃ scale, posing the risk of pipe corrosion due to its high corrosive characteristics. Thus, the purpose of this study was to determine the impact of adding EDTA additive into HCl solution during the acidifying treatment procedure. The methodology included various stages such as scale identification, chemical scale removal test using 7.5% and 15% HCL solution, 15% HCl solution test with EDTA as an additive, and corrosion determination using corrosion coupon. The results showed that 15% HCl solution was very effective in removing CaCO₃ scale but had a high corrosion rate of 186.255 mpy. Furthermore, the addition of 10 mL EDTA solution as an additive removed scale and reduced corrosion rate by approximately 85%.

Keywords: acidizing treatment; EDTA; hydrochloric acid; scale

Abstrak

Acidizing treatment adalah metode yang umum digunakan dalam mengatasi masalah scale pada peralatan produksi. HCl merupakan jenis asam yang sering digunakan dalam mengatasi scale CaCO₃. Namun penggunaan HCl dapat menyebabkan korosi pada pipa karena memiliki sifat korosif yang tinggi. Penelitian ini bertujuan untuk mengetahui pengaruh penambahan zat additive EDTA dalam larutan HCl pada proses acidizing treatment. Tahapan penelitian dilakukan meliputi identifikasi scale, uji chemical scale dispersant menggunakan larutan HCl 7,5% dan 15%, uji larutan HCl 15% yang ditambah EDTA sebagai zat additive serta identifikasi korosi dengan corrosion coupon. Hasil penelitian menunjukkan larutan HCl 15% sangat efektif dalam menghilangkan scale CaCO₃, tetapi memiliki nilai laju korosi yang tinggi sebesar 186,255 mpy. Penambahan zat additive larutan EDTA sebesar 10 mL pada metode acidizing treatment mampu menghilangkan scale CaCO₃ dan mengurangi laju korosi hingga 85%.

Kata kunci: acidizing treatment; asam klorida; EDTA; scale

Effect of EDTA Addition on Acidizing Treatment Process

1. Introduction

Crude oil is a hydrocarbon compound that produces water, oil, and gas in the production process. As these three fluids flow to the surface, friction, changes in flow rate, and pressure cause scale formation on pipe walls [1]. Scale, corrosion, and emulsion are common production problems in oil field environments [2].

The presence of a crystal-shaped scale is closely related to the content of cations and anions in water formation [3]. It can form layers capable of reducing pipe diameter, decreasing the flow rate, and causing pipe blockages. Scale also results in corrosion due to the reaction of metal with ions adhering to the surface [4], causing an increase in pressure inside the pipe, leading to rupture [5].

Prevention and reduction of scale formation can be achieved by injecting chemical substances. To address the issue of scale formation, acidizing treatment, or acid stimulation, is a commonly used method [1]. This method includes injecting specific acids into production well to dissolve deposits or inhibit crust formation [6].

Hydrochloric acid (HCl) is commonly used to address scale issues in production pipe, serving as the most common solvent to dissolve CaCO_3 scale [7]. The reaction between HCl and carbonate effectively dissolves scale [8], posing the risk of pipe corrosion due to the high corrosiveness [9]. To address pipe corrosion when using HCl in acidizing treatment, an additive is introduced to act as an inhibitor [6].

Mixtures of organic acids can be used as additives to reduce the excessive corrosiveness of HCl [10]. The addition of consists of scale and corrosion coupons,

organic substances is commonly applied to enhance the corrosion resistance of reinforced concrete in marine environments [11]. Organic compounds can also inhibit corrosion, specifically in acidic environments [12]. Previous research reported that the addition of organic substances such as oleic acid and arginine in acidizing treatment showed promising abilities in reducing corrosion rates [13][14]. However, the use of inorganic substances as corrosion inhibitors is limited due to environmental risks [15]. Organic compounds are a more environmentally friendly and efficient alternative to corrosion inhibitors in formation of adsorption films [16] [17].

The solution of Ethylene Diamine Tetraacetic Acid (EDTA) is a synthetic organic acid capable of forming bonds with various metal ions. The solution can adhere to the metal surface, acting as a coating that does not come into contact with the flow, thereby inhibiting metal oxidation and preventing corrosion [18].

Previous research showed that HCl solutions were available and efficient in addressing scale but may lead to corrosion on equipment. This limitation shows the need to investigate the effect of adding additives to acidizing treatments. Therefore, this research aimed to determine the impact of adding EDTA as an additive in acidizing treatment on scale solubility and corrosion rate in oil field production facilities.

2. Research Methods

2.1 Instruments and Materials

The instruments used in this research are corrosion and scale systems, as shown in Figure 1. Furthermore, the instrument including service valves and retrievers,

Effect of EDTA Addition on Acidizing Treatment Process

which function as the upper assembly to install and place coupons into the production pipe (flowline). Coupon holder was used to secure the voucher inside production pipe, followed by the cover for coupons and holder assembly. Other tools used include a pH meter and a DR 5000 spectrophotometer. The materials used included formation water samples, CaCO_3 scale obtained from the oil well production pipe (flowline), deionized water (aquades), NaOH, pH 10 buffer solution, EBT indicator, "Vario Ferro F10" reagent, potassium chromate indicator, AgNO_3 , reagen "Vario Sulpha 4/F10", reagent, scale dispersant (HCl), and the additive substance EDTA (Merck).

2.2 Research Stages

2.2.1 Scale Identification

Scale identification was conducted through the analysis of the ion content in formation water and measuring scales formed on production equipment. The ion content formed in formation water was analyzed using quantitative analysis, which included titrimetric and spectrophotometric analyses. The measurement was conducted by installing a scale coupon on production equipment. In the first step, the cover holder of coupon was opened and assembled with the service valve as well as the retriever. The service valve was installed at the designated point for placing coupon as presented in Figure 1. Subsequently, a coupon was placed on production pipe (flowline) for a specific period to obtain samples and scale size of the coupon.

2.2.1.1 pH Measurement

pH measurement was carried out by immersing the pH meter in formation water. Subsequently, the pH value was

read when the instrument showed a stable number.

2.2.1.2 Calcium and Magnesium Content Measurement

The measurement of calcium ions (Ca^{2+}) and magnesium ions (Mg^{2+}) was performed using titration methods. A total of 10 mL of formation water sample was placed into a 100 mL volumetric flask and diluted with deionized water to 100 mL. Subsequently, 10 mL of diluted solution was mixed with 10 drops of 1 N NaOH solution and murexide indicator. The solution was titrated with a standard EDTA 0.02 N solution to achieve a color change from pink to purple (titrant volume = A). Subsequently, 1-2 mL of pH 10 buffer solution and 30-50 mg of EBT indicator/solution were added to 10 mL diluted sample solution. The solution was titrated with a standard EDTA 0.02 N solution until a red-to-violet color change occurred, turning blue (titrant volume = B). Ca^{2+} and Mg^{2+} content were calculated using Equations 1 and 2.

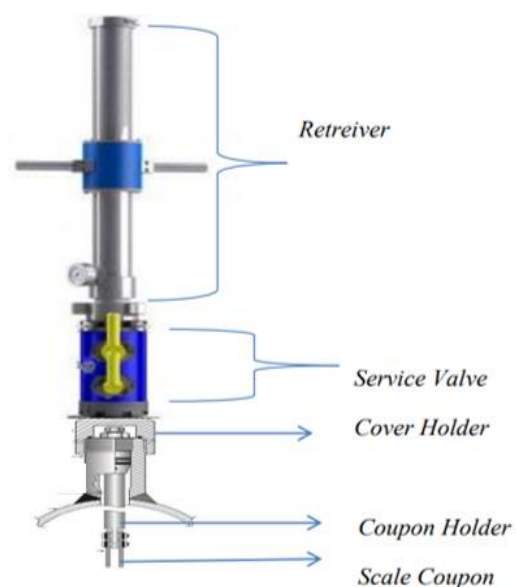


Figure 1. Corrosion and scale system

Effect of EDTA Addition on Acidizing Treatment Process

2.2.1.3 Measurement of Total Iron (Fe)

The measurement of iron ions were performed using a DR 5000 spectrophotometer. Initially, 10 mL of formation water was placed into a sample bottle with a diameter of 24 mm or 10 mm cuvette and tightly sealed. The sample bottle was placed in spectrophotometer sample slot and the instrument was calibrated by pressing the "ZERO" button. Subsequently, the sample bottle was removed, the "Vario Ferro F10" reagent was added, and it was tightly sealed and inverted to ensure a thorough mixture. This was followed by placing the sample slot and pressing the "TEST" button. After the reaction occurred for 3 minutes, the measurement results were displayed in mg/L.

2.2.1.4 Chloride Content Measurement

Chloride ion (Cl-) measurement was carried out using titration methods. Initially, 1 mL of 5% w/v potassium chromate indicator was added to the sample. This was followed by titration using the AgNO₃ solution until a color change from yellow to brick red occurred. Cl- concentration was calculated using Equation 3.

2.2.1.5 Barium Content Measurement

The measurement of barium ions was carried out using a DR 5000 spectrophotometer. A 100 mL sample of formation water was added to 5 mL of hydrochloric acid and heated until almost dry. Subsequently, the solution was diluted, and the absorbance was measured with a spectrophotometer. Barium content was calculated using Equation 4.

2.2.1.6 Sulphate Content Measurement

Sulphate ion (SO₄²⁻) was measured using DR 5000 spectrophotometer. A 10

mL of formation water was placed into a sample bottle with a diameter of 24 mm or 10 mm cuvette and tightly sealed. The sample bottle was placed in the spectrophotometer sample slot, and the instrument was calibrated by pressing the "ZERO" button. The sample bottle was removed, the "Vario Sulpha 4/F10" reagent was added, tightly sealed, and inverted to ensure thorough mixing. This was followed by placing the bottle in the sample slot and pressing the "TEST" button. After the reaction occurred for 5 minutes, the measurement results were shown in mg/L.

2.2.1.7 Sodium Content Measurement

The measurement of sodium ions (Na⁺) was carried out by comparing the difference between the total number of anions and cations. The results of anion and cation analyses in mg/L were converted to milliequivalents/L (mEq/L). The quantity of Na⁺ was obtained by subtracting the total mEq of anions from the mEq of cations. Subsequently, the mEq/L value of Na⁺ was converted to mg/L Na⁺ by multiplication with the correction factor.

$$Ca^{2+} (ppm) = \frac{AxNx40.1x1000}{v} \dots\dots\dots (1)$$

$$Mg^{2+} (ppm) = \frac{(B-A)xNx24.3x1000}{v} \dots\dots (2)$$

$$Cl^{-} (ppm) = \frac{(Vol\ titran)x(Ntitran)x35,450x1000}{v} \dots\dots\dots (3)$$

$$barium\ content\ \left(\frac{mg}{L}\right) = Cx\ fp \dots\dots\dots (4)$$

- Explanation:
- V = sample volume (mL)
 - N = normality of EDTA solution
 - A = volume of EDTA titrant required using murexide indicator
 - B = volume of EDTA titrant required using EBT indicator
 - C = concentration obtained from the measurement
 - fp = dilution factor

Effect of EDTA Addition on Acidizing Treatment Process

2.2.2 CaCO₃ Scale Dissolution Test

The scale sample used was 25 grams, with the volume of HCl solution, serving as scale dispersant, which varied at 0 mL, 10 mL, 20 mL, and 30 mL. Both components were placed in a container and soaked for 1 hour at room temperature. Subsequently, the solution was filtered to collect the undissolved scale sample. After drying, the sample was weighed, and the dispersed portion was calculated using the formula in Equation 5. The test was also conducted using a mixture of HCl solution (30 mL) and EDTA additive at concentrations of 0, 5, 10, 20, and 30 mL.

2.2.3 Corrosion Test

Approximately 25 grams of scale were weighed and placed into a test vessel using a holder containing 500 mL of dispersant solution, or approximately 2/3 of the test specimen volume. Scale dispersant solution consisted of EDTA solution with varying volumes ranging from 0 mL, 5 mL, 10 mL, 20 mL, and 30 mL, which was added to a 15% HCl solution, lasting for 24 hours. Subsequently, the test specimen was lifted, cleaned, and reweighed to determine the lost mass. The corrosion rate was calculated using Equation 6.

$$\% \text{ lost mass} = \frac{(M_0) - (M_1)}{M_0} \times 100 \dots\dots(5)$$

$$\text{corrosion rate} = \frac{(M_0 - M_1)}{A \times t \times \rho} \dots\dots\dots (6)$$

Where:

- M₀ = Initial mass
- M₁ = Final mass
- A = Surface area
- t = Time
- ρ = Density

3. Results and Discussion

3.1 Scale Identification

Scale is a solid compound originating from substantial amounts of ions present in water formation [19]. Furthermore, it disrupts the flow by reducing the inside diameter of the pipe which can hinder production and damage equipment [20].

Scale identification was conducted to understand the characteristics of scale formed inside the pipe. To measure scale growth, coupons are weighed by determining the increase in mass per unit of time. The component of water passing through the pipe generally affects scale formation, which is determined based on tests characterizing ions [8].

The results of formation water characteristics test in Table 1 showed the composition of ion content present in scale. The measurement results showed that the formation water has a pH of 7.34. This is because a higher pH increases the tendency for CaCO₃ scale formation. Based on the characteristic test, the formed scale was identified as carbonate, with a measured mass of 377.116 grams over 10 days.

Table 1. Identification test results

Parameter	mg/L	mEq/L
Ca ²⁺	236.472	11.600
Mg ²⁺	14.592	1.200
Ba ²⁺	-	-
Fe ³⁺	0.060	0.004
Na ⁺	7,828,309	340.406
Cl ⁻	11,458.187	322.794
HCO ₃ ²⁻	1,464.480	24.000
CO ₃ ²⁻	120.020	3.429
OH ⁻	0.000	0.000
SO ₄ ²⁻	163.000	3.187
pH	7.34	
scale mass	377.116 g	

Effect of EDTA Addition on Acidizing Treatment Process

3.2 Acidizing Treatment using HCl

The most commonly used acid to address the carbonate scale is HCl [16]. HCl is highly effective in dissolving carbonate scales as it reacts with carbonate formations, producing calcium chloride, carbon dioxide, and water [8]. The reaction that occurs is expressed as:



Figure 2 shows that a 7.5% HCl solution with a volume of 5 mL can dissolve 36% of CaCO₃ scale. The solubility of scale continues to increase with the addition of HCl solution volume. This phenomenon occurs because higher acid concentrations result in more hydrogen ions in the solution, enhancing the ability of HCl to dissolve scale. Consequently, a 7.5% HCl solution with 10 mL and 20 mL volumes would dissolve 52% and 64% of scale, respectively. The highest dissolution capacity was achieved with a 7.5% HCl solution at a volume of 30 mL, dissolving 84% of CaCO₃ scale.

The test results using a 15% HCl solution in the acidizing treatment process are presented in Figure 2. It was discovered that 72% of CaCO₃ scale was dissolved in a 5 mL HCl solution. The use of a 15% HCl solution in the amount of 10 mL dissolves 80% of the scale. The solubility also increased with a higher volume of 15% HCl. The increase in the concentration of hydrogen ions in the solution affected the characteristics of the chemical reaction due to the high frequency of particle collisions [21]. Moreover, soluble complex compounds tend to form as the reaction balance shifts due to adding HCl.

The solubility of CaCO₃ scale reached 100% when using 15% HCl at

volumes of 20 mL and 30 mL. Furthermore, it was discovered that the addition of acid could increase the porosity and permeability of the surface, resulting in easy solubility at high concentrations [22]. HCl solution demonstrated high effectiveness in removing CaCO₃ scale in acidizing treatment, with high dissolution power [23]. The comparison of the dissolution capabilities of 7.5% and 15% HCl solutions is shown in Figure 2. The results showed that higher concentration of HCl required a smaller volume for effective dissolution.

The results presented in Figure 2 showed that a 15% HCl solution in 20 mL and 30 mL volumes could remove 100% of the scale content within 60 minutes. The use of a 15% HCl solution in the amount of 30 mL showed a faster removal rate compared to a 20 mL solution, lasting for only 21 minutes. Consequently, the selected solution for the corrosion test was 15% HCl with a volume of 30 mL, which showed 100% performance dissolution in 21 minutes.

3.3 Acidizing Treatment using HCl and EDTA

Various acid compositions are commonly applied to enhance efficiency in acidizing treatment process [24]. Among several acid compositions, EDTA solution is a complex organic acid consisting of polycarboxylic amino acids that can be used as an additive.

Based on the test results shown in Table 2, higher content of EDTA additive in HCl solution resulted in a longer time required to dissolve scale. EDTA additive also formed complex bonds with metal ions, such as calcium-EDTA complex, which decelerated the dissolution process [25].

Effect of EDTA Addition on Acidizing Treatment Process

Previous research showed that the addition of EDTA to HCl solution could also act as an inhibitor, preventing scale formation on equipment [19]. Furthermore, the presence of additives acting as inhibitors can keep the cation-anion-forming scales in the solution [26]. For example, tartaric acid, when used as an additive, shows the ability to inhibit the growth of barium sulphate as the crystal structure formed causes difficulty in adhering to the equipment [6].

Based on the results of the 15% HCl and EDTA solution, corrosion rate was tested by adding EDTA solution with volumes of 0 mL, 5 mL, and 10 mL. This selection was influenced by the ability of the solution to achieve 100% performance in the dissolving scale at a relatively fast time, as shown in Table 2.

3.4 Measurement of Corrosion Rate

The speed of material quality deterioration over time can be expressed as a corrosion rate. The resistance level of material to corrosion generally has a corrosion rate value ranging from 1-200 mils per year (mpy) [27]. In this research, corrosion rate measurements using a 15% HCl solution, as shown in Figure 3, had a value of 186.255 mpy. Among the five levels of material resistance to corrosion, Mars G. Fontana (1987) in Afandi et al. [27] classified a range of 50-200 mpy in the poor corrosion resistance category, indicating a very high corrosion rate on the equipment. Generally, corrosion occurs in pipes due to a high acid concentration [1][28].

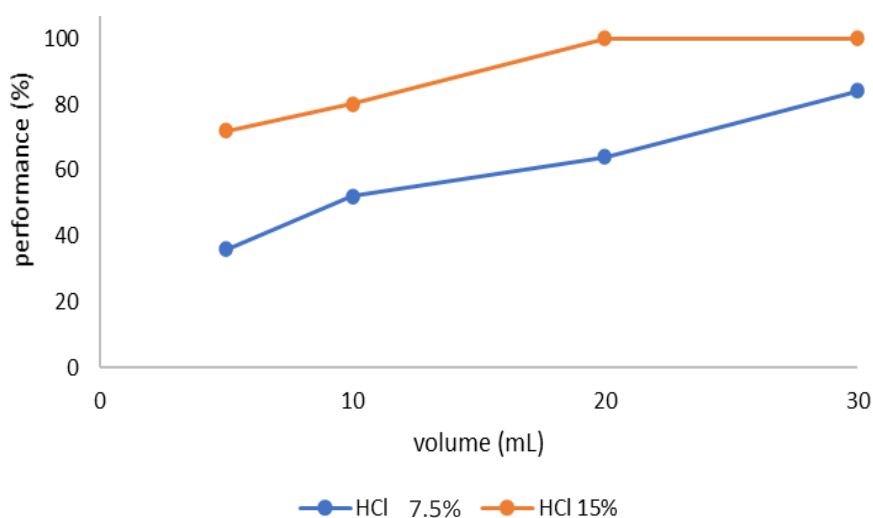


Figure 2. Scale solubility in HCl solution

Table 2. Test results of 15% HCl solution and EDTA additive

No	Solution Mix		CaCO ₃ Scale			Total Time (minute)
	HCl 15% (mL)	EDTA (mL)	Initial Scale (g)	Final Scale (g)	Performance (%)	
1	30	0	25	0	100	21
2	30	5	25	0	100	30
3	30	10	25	0	100	33
4	30	20	25	0	100	39
5	30	30	25	0	100	46

Effect of EDTA Addition on Acidizing Treatment Process

Based on Figure 3, corrosion rate measurements for the mixture of 15% HCl and 5 mL EDTA showed a value of 46.754 mpy, belonging to the fair category. Meanwhile, the mixture of 15% HCl and 10 mL EDTA has a corrosion rate of 33.467 mpy, which categorizes it as corrosion-resistant (fair). The corrosion rate decreases by approximately 85% compared to the rate without using the EDTA additive.

The addition of EDTA additive in HCl solution reduced the corrosivity of HCl. This reduction was attributed to the numerous oxygen and nitrogen atoms in EDTA capable of binding with metal ions. EDTA additives can form complex compounds with metal ions in corrosion, specifically by binding with Ca^{2+} , Fe^{2+} , and Fe^{3+} ions [20].

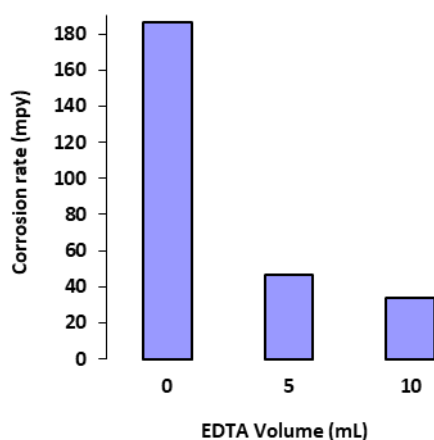


Figure 3. Corrosion rate measurement for a 15% HCl solution and EDTA addition

The research by Fouda et al. [29] showed that the addition of organic substances in HCl reduced the corrosion rate of zinc because the thin layer produced could isolate the metal surface. The use of oleic acid in the acidizing treatment process showed a decreased corrosion rate from 2,525.12 to 77.34 mpy [13]. The addition of other additives, such as arginine, to the HCl solution, also reduced the corrosion rate. This significant reduction occurred due to the function of arginine, acting as an inhibitor, and forming a passive layer on the surface, which obstructed the attack of corrosive solutions [14].

4. Conclusion

In conclusion, this research showed that acidizing treatment using a 15% HCl solution was highly effective in removing CaCO_3 scale. However, it had a corrosion rate of 186.255 mpy, which was categorized as poor. The addition of an EDTA additive solution at 10 mL in a 30 mL HCl solution in acidizing treatment method effectively eliminated the CaCO_3 scale and reduced the corrosion rate by 85%.

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Effect of EDTA Addition on Acidizing Treatment Process

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