Study on the Addition of Ethylene Diamine Tetra Acetic Acid (EDTA) as Inhibitor Calcium Sulfate ($CaSO_4$)

Nurul Rosadinah[1], Suharso[2], Agung Abadi Kiswandono[2]

[1]Postgraduated student of Chemistry, Faculty of Mathematics and Natural Sciences, University of Lampung, Bandar Lampung, 35145, Indonesia

[2]Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Lampung,Bandar Lampung, 35145, Indonesia

nurulrosadinah28@gmail.com

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Abstract: In the oil and gas industry, a common problem is salt deposition on the walls of fluid flow processing equipment, especially on heat transfer surfaces and on the surfaces of evaporation equipment. This deposition is undesirable because it causes scale formation which can affect heat transfer as well as reduce efficiency and prevent flow in the process fluid. To overcome this, this research was used ethylene diamine tetra acetic acid (EDTA) as inhibitor calcium sulfate ($CaSO_4$). The aim of the present study was to determine the effectiveness of EDTA in inhibiting the formation of calcium sulfate ($CaSO_4$) scale.

Ethylene diamine tetra acetic acid (EDTA) as inhibitor calcium sulfate ($CaSO_4$) with methods unseeded experiment and seeded experiment at temperature of 90 ℃ with variations in the concentration of the growth solution 0.1; 0.15; and 0.2 M and variations in inhibitor 5, 15, 25, 35, dan 45%. Results: Percent effectiveness value obtained at growth solution concentration of 0.1; 0.15; 0,2 M and inhibitor concentration of 5% using the unseeded experiment method was 86.296%; 77.435%; 68.354%. Percent effectiveness value obtained at growth solution concentration of 0.1; 0.15; 0.2 M and inhibitor concentration of 5% using the seeded experiment method was 89.385%; 38.182%; 62.532%. The most effective percent effectiveness value obtained at growth solution concentration of 0.1 M and inhibitor concentration of 5% using the unseeded experiment method was 86.296%. The most effective percent effectiveness value obtained at growth solution concentration of 0.1 M and inhibitor concentration of 5% using the seeded experiment method was 89.385%.

1 INTRODUCTION

Most industrial processes face quite significant problems is salt deposition on the walls of fluid flow processing equipment, especially on heat transfer surfaces and on the surfaces of evaporation equipment. This deposition is not expected because the precipitate causes crust formation which can affect heat transfer and reduce efficiency and prevent flow in process fluids. In addition, scale builds up in the duct pipes, holes, and some flow parts during the fluid flow process can cause serious operational problems, as this scale can cause corrosion and damage to production process equipment. Therefore, costs and the losses incurred are very high, due in large part to maintenance costs equipment is spent on replacing or repairing damaged components scale.

Scale formation can be prevented by adjusting the pH value with acid injection such as sulfuric acid or hydrochloric acid (Suharso and Buhani, 2012). This opinion in line with Suharso et al. (2013) who reported scale deposits in industry prevented by this method by lowering the pH of the solution with adding acids and using inorganic compounds (Suharso, 2013). But based on Lestari's research (2008), uses acid to prevent

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scale formation, it is also not effective in high concentrations because it can increase speed corrosion and conductivity, and has a fairly high level of danger. Business another thing used by Halimatuddahliana (2003) is to prevent scale formation by softening and freeing water minerals, the weakness of this method is need a lot of money. A method was developed based on some of these possible weaknesses used to prevent or control the problem of scale formation by adding inhibitors. One of the methods used to inhibit scale formation by adding inhibitors to the scale. Inhibitors are substances used to control scale growth with the aim of reducing, preventing, or delaying crust formation. Types The inhibitor that are often used are weak acid compounds (carboxylic acids) and metal ions. Examples of weak acids (carboxylic acids) used as inhibitors including citric acid, tartaric acid, and malic acid while metal ions are used as an inhibitor are copper ions (Cu^{2+}) (Rabizadeh, 2014). Selecting the right inhibitor can provide cheap and effective reduction effective in scale formation as low concentrations can have an impact great effect on crystal growth.

Organic or inorganic molecules with chelating properties are used as inhibitor to prevent scale such as including ethylene diamine tetra acetic acid (EDTA), ethylene diamine tetra methylene phosphonic acid (EDTMP), amino trimethylene phosphonic acid (ATMP), dan 1-hydroxyethylidene-1,1-diphosphonic acid (HEDP) (Khormali, 2016). Function agent chelator is for prevent scale formation. Part anions from inhibitors join with part cations, for example ions metal in solution produce formation bond coordination with cations from scale (Kumar, 2018). As typical chelate, EDTA is kind ligand chelation with high affinity which can forming complex metal−EDTA.

2 MATERIALS AND METHOD

2.1 Materials and Equipments

Glassware used in laboratories, water bath, plastic cups, magnetic stirrer, spinbar, oven, analytical balance, and pH meter. Calcium chloride ($CaCl2$), sodium sulfate ($Na2SO4$), distilled water, EDTA and filter paper.

2.2 Making EDTA Inhibitors

In this study, EDTA was used with varying concentrations of 5, 15, 25, 35, and 45%. Making an inhibitor solution with a concentration of 5% is carried out by take 5 g of EDTA, then dilute it with distilled water in a 100 mL volumetric flask, then homogenized. The same treatment is carried out to prepare the solution inhibitors with concentrations of 15, 25, 35, and 45%.

2.3 Seed Crystal Production

Crystal seeds were made from 27.75 g of CaCl2 dissolved in a volume of distilled water with total volume of 250 mL was then stirred using a magnetic stirrer at a temperature of 90 ℃ for 15 minutes and Na2SO4 35.5 g dissolved in distilled water with total volume of 250 mL was then stirred with a magnetic stirrer at 90 ℃ for 15 minute. The Na2SO4 and CaCl2 solutions were mixed and stirred with a magnetic stirrer at a temperature of 90℃ until it settles completely. Then the precipitate is separated with filter paper and the precipitate is dried using an oven at temperature 105℃.

2.4 Experiments of Crystallization

a. Unseeded Experimental Method

The growth solution was prepared from 0.1 M $CaCl_2$ solution and 0.1 M Na2SO4 solution each with a total volume of 300 mL EDTA inhibitor with a predetermined concentration. Each solution put into a beaker and stirred using a magnetic stirrer 15 minutes at 90 °C to homogenize the solution. Then solution 0.1 M CaCl2 and 0.1 M Na_2SO_4 solution were mixed and stirred using a magnetic stirrer for 15 minutes at 90°C to form $CaSO_4$ scale and the pH value was measured using a pH meter.

The CaSO4 solution formed was put into 5 plastic cups each as much as 50 mL. After that, place it in a water bath at 90 °C for 15 minutes to reach equilibrium. Observations were carried out for 60 minutes, at for the first 20 minutes, one glass is taken, then filtered using filter paper which has been weighed, then dried using an oven at a temperature of 105 °C for 3-4 hours. Then the glass is taken again every 10 minutes until the the last glass. This experiment was repeated with varying concentrations of the CaCl2 solution and Na2SO4 of 0.15 and 0.2 M as well as varying the concentration of the EDTA inhibitor with a predetermined concentration.

b. Seeded Experimental Method

The growth solution was prepared from 0.1 M $CaCl_2$ solution and 0.1 M Na_2SO_4 solution each with a total volume of 300 mL EDTA inhibitor with a predetermined concentration. Each solution put into a beaker and stirred using a magnetic stirrer 15 minutes at 90 °C to homogenize the solution. Then solution 0.1 M $CaCl_2$ and 0.1 M Na_2SO_4 solution were mixed and stirred using a magnetic stirrer for 15 minutes at 90°C to form $CaSO_4$ scale and the pH value was measured using a pH meter.

The CaSO4 solution formed was put into 5 plastic cups contains 0.2 grams of crystal seeds, 50 mL each. After that, place it in a water bath at 90 °C for 15 minutes to reach equilibrium. Observations were carried out for 60 minutes, at for the first 20 minutes, one glass is taken, then filtered using filter paper which has been weighed, then dried using an oven at a temperature of 105 °C for 3-4 hours. Then the glass is taken again every 10 minutes until the the last glass. This experiment was repeated with varying concentrations of the $CaCl_2$ solution and Na_2SO_4 of 0.15 and 0.2 M as well as varying the concentration of the EDTA inhibitor with a predetermined concentration.

2.5 Data Analysis

The data produced as amount of precipitate versus time at various concentrations of growth solution and in the presence of additive, each was plotted as an amount of precipitate versus time using MS Excel.

3 RESULTS AND DISCUSSION

The effect of adding EDTA varies concentration inhibits the formation of $CaSO_4$ deposit in growth solution concentrations of 0.1, 0.15, and 0.2 M shown in Fig 1-3. At $CaSO_4$ growth solution concentrations of 0.1 M (Figure 1), EDTA was only ableto inhibit its growth from CaSO4 crystals are close to 100%. At $CaSO_4$ growth solution concentrations of 0.15 M (Figure 2) and 0.2 M (Figure 3), EDTA not only completely inhibited the growth rate of $CaSO_4$ crystal.The lowest inhibitor effectiveness occurred at the concentration of growth solution with a dose of the EDTA added at 25-45%.

$$\text{Effectiveness of inhibitor EI (\%)} = 100\ x\ \frac{(Ca - Cb)}{(Cc - Cb)}$$

where Ca = the amount of $CaSO_4$ precipitation in the presence of EDTA at equilibrium (g); Cb = the amount of $CaSO_4$ precipitation in the absence of liquid smoke at equilibrium (g); Cc = the amount of $CaSO_4$ precipitation in the initial state(g).

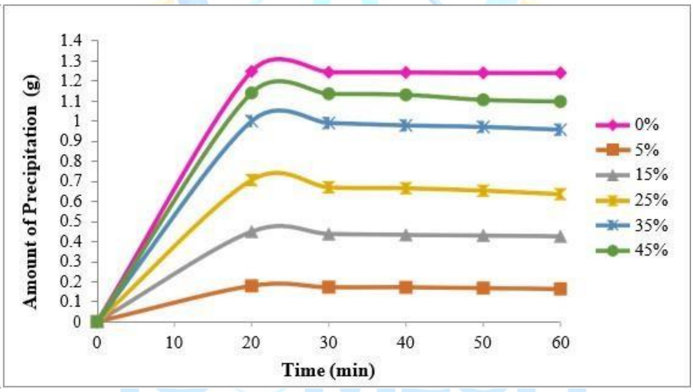

Figure 1: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitor concentrations at the growth solution concentration of 0.1 M unseeded experimental method.

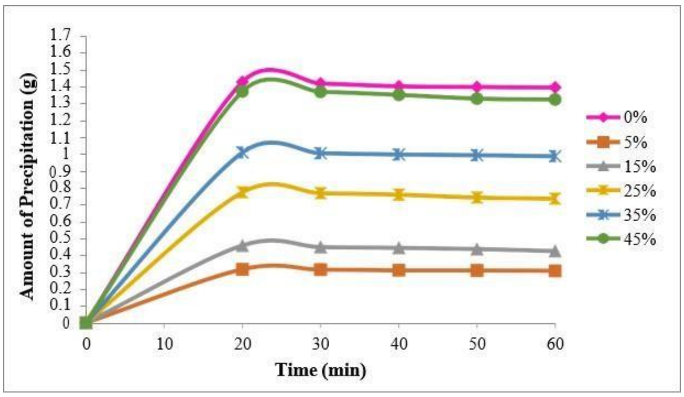

Figure 2: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitorconcentrations at the growth solution concentration of 0.15 M unseeded experimental method.

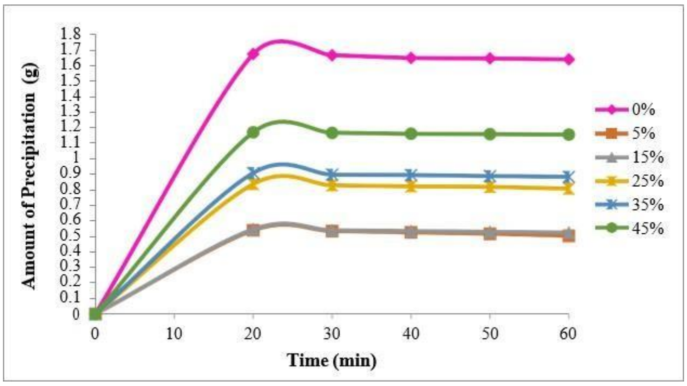

Figure 3: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitorconcentrations at the growth solution concentration of 0.2 M unseeded experimental method.

Table 1: Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.1 M unseeded experimental method.

No.	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	86.269
2.	15	64.996
3.	25	46.401
4.	35	21.191
5.	45	9.711

Table 2: Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.15 M unseeded experimental method.

No.	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	77.435
2.	15	68.554
3.	25	46.325
4.	35	29.086
5.	45	4.211

Table 3: Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.2 M unseeded experimental method.

No.	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	68.354
2.	15	67.713
3.	25	50.306
4.	35	45.955
5.	45	29.777

As seen in Table 1-3 and Figure 1-3, the inhibition of $CaSO_4$ scale formation substantially decreased as the inhibitor concentration increased. Thus, the percentage efficiency of the inhibitor decreases when the inhibitor concentration level increases from 5 to 45%. The effect of adding EDTA varies concentration inhibits the formation of $CaSO_4$ deposit in growth solution concentrations of 0.1, 0.15, and 0.2 M shown in Figure 4-6. At $CaSO_4$ growth solution concentrations of 0.1 M (Figure 4), EDTA was only ableto inhibit its growth from CaSO4 crystal seeds are close to 100%. At $CaSO_4$ growth solution concentrations of 0.15 (Figure 5) and 0.2 M (Figure 6), EDTA not only completely inhibited the growth rate of $CaSO_4$ crystal seeds but is also capable of dissolving $CaSO_4$ crystal seeds.

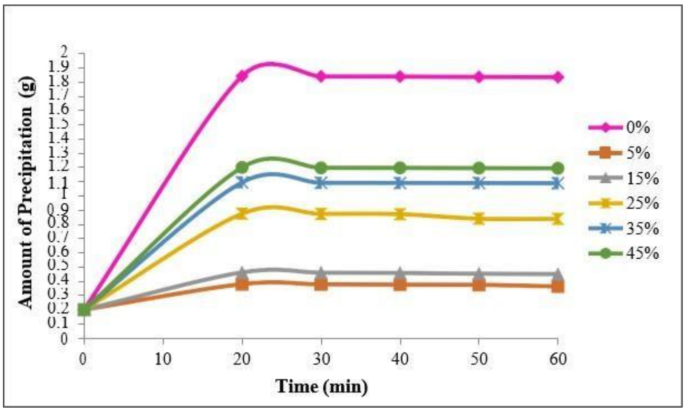

Figure 4: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitor concentrations at the growth solution concentration of 0.1 M seeded experimental method.

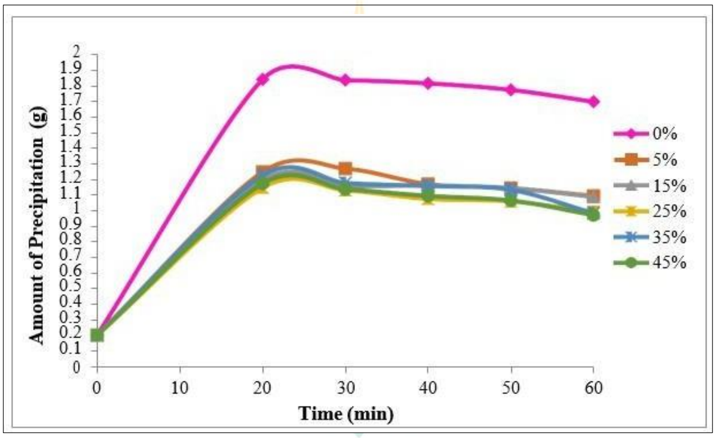

Figure 5: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitor concentrations at the growth solution concentration of 0.15 M seeded experimental method.

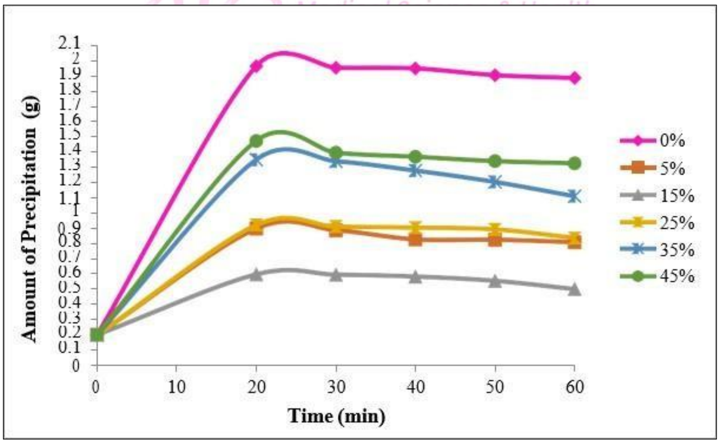

Figure 6: Amount of $CaSO_4$ precipitation vs. time with the addition of various inhibitor concentrations at the growth solution concentration of 0.2 M seeded experimental method.

Table 4: Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.1M seeded experimental method.

No	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	89.385
2.	15	84.333
3.	25	59.736
4.	35	45.607
5.	45	39.165

Table 5. Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.15 M seeded experimental method.

No	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	38.182
2.	15	40.439
3.	25	44.721
4.	35	41.297
5.	45	44.239

Table 6. Effectiveness of inhibitor in inhibiting the scale formation of $CaSO_4$ at the growth solution concentration of 0.2 M seeded experimental method.

No	Adding of Inhibitor (%)	Effectiveness of Inhibitor (%)
1.	5	62.532
2.	15	78.867
3.	25	59.987
4.	35	39.007
5.	45	31.796

As seen in Table 4-6 and Figure 4-6, the inhibition of $CaSO_4$ scale formation substantially decreased as the inhibitor concentration increased. Thus, the percentage efficiency of the inhibitor decreases when the inhibitor concentration level increases from 5 to 45%. The effect of adding EDTA varies concentration inhibits the formation of $CaSO_4$ deposit in growth solution concentration of 0.1, 0.15 and 0.2 M shown in Fig. 4-6. At $CaSO_4$ growth solution concentration of 0.1 M (Figure 4), EDTA was only able to inhibit its growth from $CaSO_4$ crystal seeds are clode to 100%. At CaSO4 growth concentration of 0.15 and 0.2 M, the inhibitor's ability the formation of $CaSO_4$ crystals will be slower than at $CaSO_4$ growth concentration of 0.1M. Even in these situations, the EDTA can dissolve the $CaSO_4$ crystal seeds that are available. Increasing the concentration of $CaSO_4$ growth solution and decreasing the dose of liquid smoke added to the growth solution will gradually reduce the ability of the EDTA to inhibit the growth of $CaSO_4$ crystal seeds. The lowest inhibitor effectiveness occurred at the concentration of 0.2 M growth solution with a dose of the EDTA added at 45% which was 31% (Table 6).

4 CONCLUSION

Inhibitor of EDTA can inhibit the formation of calcium sulfate ($CaSO_4$) scale crystals. The ability of the EDTA to inhibit the formation of calcium sulfate ($CaSO_4$) scale was most effective in growth solution unseeded experiment with a concentrationof 0,1 M and at concentration of 5% inhibitor with an effectiveness percentage value of 86,269 %. The ability of the EDTA to inhibit the formation of calcium sulfate ($CaSO_4$) scale was most effective in growth solution seeded experiment with a concentration of 0,1 M and at concentration of 5% inhibitor with an effectiveness percentage value of 89,385 %.

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