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The Performance of Sulfamic, Acetic, and Citric Acid as An Alternative Carbonate Scale Dissolution at Laboratory Scale

Meyliana Wulandari[1], Verawati[2], Isalmi Aziz[2], Nofrizal[3], Rachmi Kartini[3],
and Pandian Bothi Raja[4]

[1]Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Negeri Jakarta
R. Mangun Muka Raya Street, Rawamangun, Pulo Gadung Subdistrict, East Jakarta City, 13220, Indonesia.

[2]Department of Chemistry, Faculty of Science and Technology, State Islamic University Syarif Hidyatullah Jakarta
Ir H. Juanda Street No. 95, Ciputat Timur District, South Tangerang City, Banten 15412, Indonesia.

[3]Testing Center for Oil and Gas LEMIGAS
Ciledug Raya Street Kav.109, Cipulir, Kebayoran Lama, South Jakarta 12230, Indonesia.

[4]School of Chemical Sciences, Universiti Sains Malaysia
Minden, 11800 Penang, Malaysia

Corresponding author: meyliana.wulandari@unj.ac.id

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ABSTRACT - Calcium carbonate scale is being considered one of the challenges being faced by the oil and gas industry, as it is disrupting production activities. Acidification using hydrochloric acid is being widely applied as a scale remover; however, this reagent is being recognized as highly corrosive. Hence, this study is aiming to investigate the effect of combining sulfamic acid, citric acid, and acetic acid in ratios of 9:3:3, 6:6:3, and 3:9:3 at temperatures of 35°, 45°, and 50 °C on an environmentally friendly scale dissolution. The percentage of scale dissolution from the three acids is being compared with 5, 10, and 15% hydrochloric acid at temperatures of 35°, 45°, and 50°C using the gravimetric method. The results of the study on a laboratory scale are showing that the largest percentage scale solubility is being obtained with a ratio of sulfamic acid: citric acid: acetic acid of 9:3:3 of 65.79% at a temperature of 50°C. When compared with 5% HCl at 50°C as a control, scale dissolution is reaching 63.49%. This result is being supported by scanning electron microscope data, which are showing that the surface of the scale on the pipe after treatment with three acid combinations is having a smoother surface morphology compared to using commercial scale removal (HCl). The use of three acid combinations in this study is serving as an alternative to commercial-scale removal.

Keywords: acetic acid, $CaCO_3$, citric acid, scale, sulfamic acid.

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INTRODUCTION

Oil and gas mining uses formation water injected into the reservoir to maintain the pressure. The aim is to ensure reservoir recovery remains high (Khormali & Petrakov 2016). Combining several minerals in the water flow (seawater and reservoir formation water) generates salt deposition (X. Liu et al. 2024). Inorganic salt deposition occurs during the formation, wellbore, and pipelines in the well, as well as in the tubing, casing, flow line, manifold, separator, tank, and inner surface of oilfield equipment (including pipelines and valves) during the production process at all well operations (Gamal et al. 2021). Formation water is water that is naturally contained in rock reservoirs (Jambak & Mahendra 2023). The oilfield scale usually contains $CaSO_4$ (anhydrite, gypsum), $BaSO_4$ (barite), $SrSO_4$ (celestite), and $CaCO_3$ compounds (Naseri et al. 2015). Calcium carbonate ($CaCO_3$) is one of the most frequently occurring scales in the oil and gas field (Man et al. 2022). $CaCO_3$ is a solid form of calcite (low-temperature T < 30 °C), and aragonite (high- temperature T > 70 °C) (Basheer et al. 2021).

The CO_2 gas in water causes an acidic atmosphere:

$$CO_2 + H_2O \qquad H_2CO_3 \tag{1}$$

When HCO_3- reacts with Ca^{2+} in water to form $CaCO_3$ (Aziz et al. 2024). Then, $CaCO_3$ experiences crystallization, aggregation, and precipitation to form white crystals (Akhdan et al. 2022). Scale blocks over the pipelines in partial or total, decreased heat transfer, energy consumption, reduced production output in the oil and gas industry, shutdowns, and economic loss (Sousa et al. 2016). The oil company in Indonesia (PT. PERTAMINA) spends 6-7 million USD every ten years to regenerate the pipeline (Suharso et al. 2017).

Ways to control scale on downhole equipment and production facilities include prevention methods with scale inhibitors (Mpelwa & Tang 2019). Previous research utilized polymaleic acid (PMA) and successfully inhibited the $CaCO_3$ scale at 40 °C, pH 8.50, and after 20 hour of incubation (Chew & Mat 2015). Organophosphate organic scale inhibitors have been reported to successfully form complexes with metals that are easily soluble in water (Demadis & Lykoudis 2005). Scale inhibitors using chelating agents are expensive (Smith et al. 2000). Therefore, affordable scale removal is still being studied to remove formed scales.

Acidizing using halide acid (hydrochloric acid (HCl) or a mixture of hydrofluoric acid (HF) and HCl is a good chemical dissolver for carbonate scale treatment because of its affordable price. The dissolving power of the scale is respectively HCl >HNO_3>HCOOH>H_2SO4 (Suleman Tahir & Saleem 2007). However, halide acid can damage metal surfaces because of its corrosive nature (Man et al. 2022). The application of HCl at high temperatures accelerates corrosion in pipelines (Zhai et al. 2020). Acetic acid, formic acid, maleic acid, and citric acid are organic acids that are less corrosive scale dissolvers when used at high pressure and high temperature (Murtaza et al. 2022).

Organic acids have a lower level of corrosion effect. The acetic and formic acid mixture at 175°C (High Pressure, High Temperature) has successfully dissolved a scale equivalent to 15% HCl at room temperature. Carboxylic acids (citric, maleic, and tartrate) at concentrations of 0-20 ppm have been shown to reduce calcium sulfate scale and are less corrosive to pipelines (Rabizadeh et al. 2014). Previous research has reported that a mixture of 10% acetic acid with 20% authigenic acid resulted in a significant increase of 6.08% in the $CaCO_3$ dissolution rate (Duan et al. 2023). Acetic acid has been reported to be a good $CaCO_3$ dissolution (Kim & Yoon 2021). The combination of citric acid and sulfamic acid, along with the scale inhibitor hexamine, has been reported to remove scale up to 100% at 65°C (Majeed 2010). Other studies have reported the utilization of scale removal from the synthesis of glucose and oleic acid with sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and para-toluene sulfonic acid catalysts (Fadillah 2020). The scale of the synthesis results was compared with commercial scale removal using HCl, H_2SO_4, and HF. The results of organic scale removal addiction to samples Silicate 07, Silicate-L1, and Carbonate-A3 with % weight reduction were 12.884%, 15.047%, and 31.909%, respectively, at a test temperature of 100°C. From these results, it was concluded that the weight reduction was not optimal. To maximize scale dissolution, the combination of sulfamic, citric, and acetic acids, which has not been previously reported, will be studied in this study.

Hence, this research aims to investigate the effects of using a mixture of organic acids (sulfamic, citric, and acetic acids) on achieving a higher $CaCO_3$ dissolution rate at varying temperatures (35, 45, and 50°C) and concentrations (9:3:3, 6:6:3, and 6:3:6%).

Cation concentration analysis was carried out using inductively coupled plasma-optical emission spectroscopy (ICP-OES) and gravimetric analysis to identify carbonates. The scale dissolver effectiveness test was then compared with HCl (5%, 10%, and 15%) and temperature (35, 45, and 50 °C), carried out gravimetrically. Surface morphology and elemental analysis were conducted using a scanning electron microscope with an energy-dispersive X-ray detector to examine the differences in the morphological shape of scales dissolved with sulfamic acid, citric acid, acetic acid, and HCl.

METHODOLOGY

Materials

The material used is a scale sample from field X1, Indonesia (Figure 1). All chemicals used were obtained from Merck, Germany, including hydrochloric acid (HCl, purity 99%), nitric acid (HNO_3, purity 99%), toluene, acetone, and methanol (for cleaning the scale from oil, water, and other impurities). Citric acid, sulfamic acid, acetic acid, millipore filter paper 0.45 µm and sodium carbonate (Na_2CO_3, purity 99%). Ultra High Purity (UHP) grade oxygen gas (O_2), argon (Ar), and nitrogen gas (N_2) were supplied by Samator Indogas, Indonesia.

Methods

Identification of Cation Concentration on Scale using ICP-OES (Perkin Elmer Optima 8300) based on American Society for Testing and Materials (ASTM) UOP 714-07

Clean and dry porcelain cups are weighed (Wo). 1 g of scale sample is weighed (Ws) and then placed in a furnace (Barsnstead, USA) at 450–500°C for one hour and 750°C for the next hour. The porcelain cup and the remainder of the annealing sample are cooled and weighed as the final weight (Wa). The resulting annealing sample is digested in HCl and nitric acid (3:1 v/v) and then heated until the dissolution is complete. The filtrate is then collected, and the metal is analyzed using ICP-OES (Perkin Elmer Optima 8300, USA). The metal concentration samples are measured by comparing them to standard solutions.

Carbonate Concentration Analysis Using the Gravimetric Method

A total of 10 grams of scale sample was acidified with 50 mL of HCl, placed in a heat-resistant glass, and crushed. Carbonate on the scale and HCl will produce gas bubbles. The gas bubbles disappear when the destruction is complete. In the next stage, the remaining crust samples mixed with HCl were rinsed using distilled water, dried in an oven (Memmert, Germany), and weighed and calculated using the following equations (2 and 3) (Marija et al. 2019).

$$\%\ CaCO_3 = \frac{\text{Sample weight - (Final weight - Initial weight)}}{\text{Sample weight}} \text{ x } 100\% \quad (2)$$

$$\%\ CO_3^{2-} = \frac{\text{Equivalent weight } CO_3^{2-}}{\text{Equivalent weight CaCO3}} \text{ x } 100\% \quad (3)$$

Calcium carbonate scale dissolution analysis using gravimetric

The calcium carbonate scale particles were crushed and sieved until a particle size of 20 mesh was obtained. The samples were dried in an oven at 60°C for 24 hours (Duan et al. 2023). Five grams of scale sample was dissolved in 50 mL of acid solvent (sulfamic acid, acetic acid, and citric acid with concentrations in the ratio (9:3:3, 6:6:3, 3:9:3) for 60 minutes at 35, 45, and 50°C). The % scale dissolution for those acids was compared with 5, 10, and 15% HCl at 35, 45, and 50°C. The remaining scale deposit is filtered using a sieve whose empty mass has been weighed. It was dried at 105°C, cooled, weighed as the final weight, and calculated using equation (4).

$$\%\ \text{scale dissolution} = \frac{\text{Sample weight - (Final weight - Initial weight)}}{\text{Sample weight}} \text{ x } 100\% \quad (4)$$

Scanning electron microscope-energy dispersive X-ray (SEM-EDX)

SEM-EDX analysis was carried out using the Philips-XL30 instrument and Oxford Instrument's Aztec software for data analysis. However, the sample must first be rinsed with isopropanol and dried.

RESULT AND DISCUSSION

Identification of Cation from the Scale

The $CaCO_3$ scale ash was obtained via destruction using nitric acid and hydrochloric acid (3:1 v/v). Nitric acid is an oxidizer, while hydrochloric acid accelerates the dissolving process dissolution and heating speeds up the dissolution. ICP-OES data shows the percentage concentration of the scale composition (Table 1).

Figure 1. Scale sample

Table 1 shows that the highest scale is calcium (Ca) at 49.41%. The high concentration of Ca^{2+} is commonly caused by the formation of water produced by oil and gas (L. Liu et al. 2021). The second highest element, namely Iron (Fe) at 0.53%, probably comes from $Fe(OH)_3$, FeOOH, and Fe_2O_3 due to corrosion on metal surfaces (Song & Chen 2015), the release of rock fragments such as siderite ($FeCO_3$) from rock surfaces and accumulation in crustal deposits and saltwater reservoir. Mg^{2+} is the third highest scale component at 0.52%, probably originating from dolomite ($CaMg(CO_3)_2$) (Kaczmarek et al. 2017). In the following order, barium (Ba) is at 0.41%. Other elements such as Strontium (Sr), Phosphorus (P), and Potassium (K) can be caused by the formation of water and Manganese (Mn) from corroded production equipment.

Gravimetric measurement to analyze the carbonate concentration

The determination of carbonate was carried out using the gravimetric method. The CO_3^{2}- concentration obtained was 49.75%, which proves that the dominant scale contains carbonate. In the formation water, carbonate in scale can be obtained from carbonate ions (CO_3^{2}-) or bicarbonate ions (HCO_3^{-}). Calcium has a larger composition than iron. The reaction between calcium ions (Ca^{2+}) and carbonate ions (CO_3^{2}-) or bicarbonate ions (HCO_3^{-}) has been confirmed to produce a calcium carbonate ($CaCO_3$) scale (Usamah & Rizal 2019).

Table 1. The cation concentration of scale using ICP-OES

No	Cations	%
1	Potassium (K)	0,01
2	Sodium (Na)	0,03
3	Calcium (Ca)	49,41
4	Magnesium (Mg)	0,52
5	Iron (Fe)	0,53
6	Strontium (Sr)	0,13
7	Barium (Ba)	0,41
8	Aluminium (Al)	<0,01
9	Silica (Si)	0,01
10	Manganese (Mn)	0,04
11	Argentum (Ag)	<0,01
12	Phosphor (P)	0,02
13	Cuprum (Cu)	<0,01

Scale dissolution analysis using gravimetric method

Figure 2 shows the relationship between acid mixture ratio, temperature, and percentage of dissolved scale (%). The sulfamic acid, citric acid, and acetic acid ratios used in this study were 9:3:3, 6:6:3, and 3:9:3. The total acid ratio used was 15%. It refers to the technical application in the field, which has been using 15% HCl to handle reservoir problems containing carbonate (Paydar et al. 2023). At this ratio, the concentration of sulfamic acid was reduced from 9.6 to 3%. It was done to avoid corrosion because the higher the concentration of sulfamic acid, the higher the corrosion rate.

Meanwhile, the citric acid ratio in this study increased from 3.6 to 9% because the increase in citric acid concentration did not significantly affect the corrosion rate. Meanwhile, the acetic acid concentration was maintained at 3% to prevent deposits from forming in contact with the scale. Previous studies have reported that acetic acid can form reaction products such as calcium acetate, which can precipitate due to its limited solubility (Kamal et al. 2018a).

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Figure 2 shows the relationship of % scale dissolution of the combination of the three acids. At a ratio of sulfamic acid: citric acid: acetic acid 3:9:3 at a temperature of 35°C, % scale dissolution was obtained at 47.90%. The ability to dissolve scale increases with increasing temperature. It can be seen that when the temperature is increased to 40°C, the precentage scale dissolution increases to 49.70%, and when the temperature is 50°C, 51.38% scale dissolution is obtained. This can be explained by the fact that the higher the temperature, the more kinetic energy is provided to the molecules, which can cause the molecules to move faster and collide more often, causing the rate of scale dissolution reaction to increase. Meanwhile, sulfamic acid: citric acid: acetic acid (6:6:3) at 35°C produced 53.55% in the second acid mixture ratio. There was an increase in the scale dissolution, when the temperature was increased to 45°C by 55.78°C and 56.55% by 50°C. Compared with the first sulfamic acid ratio (3:9:3) and the second (6:6:3), it can be seen that the higher the concentration of sulfamic acid in the mixture, the greater the % scale dissolver will be. The higher the acid concentration, the greater the H+ that will be donated to dissolve the scale. The third acid mixture ratio, sulfamic acid: citric acid: acetic acid (9:3:3) at 35°C, produced a % scale dissolver of 61.89%. Moreover, it increased to 64.40 at 45°C, and %scale dissolution reached a maximum when the temperature was increased to 50°C by 65.79%. Based on the three acid ratios, the higher the concentration of sulfamic acid, the greater the scale dissolution ability compared to increasing the concentration of citric acid. Sulfamic acid has a better dissociation rate than citric and acetic acids. In addition to being weakly dissociated, citric acid and acetic acid can also produce compounds with low solubility, such as calcium citrate and calcium acetate, so citric acid and acetic acid are less suitable for dealing with scales containing calcium. However, citric acid can be used as a dissolving agent for types of scales containing iron (Alhamad et al. 2020).

These results are supported by previous research that sulfamic acid (3, 6, and 9%) at temperatures (25, 45 and 65°C) can dissolve 65-100% of scale, and citric acid (3, 6, and 9%) at temperatures (25, 45 and 65°C) can dissolve 60-95% of scale (Majeed 2010). An increase in acid concentration causes an increase in the concentration gradient between the solution and the solid interface. It causes the scale dissolution reaction rate to increase.

Figure 3 shows the effect of hydrochloric acid concentration and temperature on the percentage scale dissolver. As the concentration of the acid solvent increases to 5, 10, and 15%, the solubility of the scale also increases. Increasing the solubility of the scale with increasing concentration is most

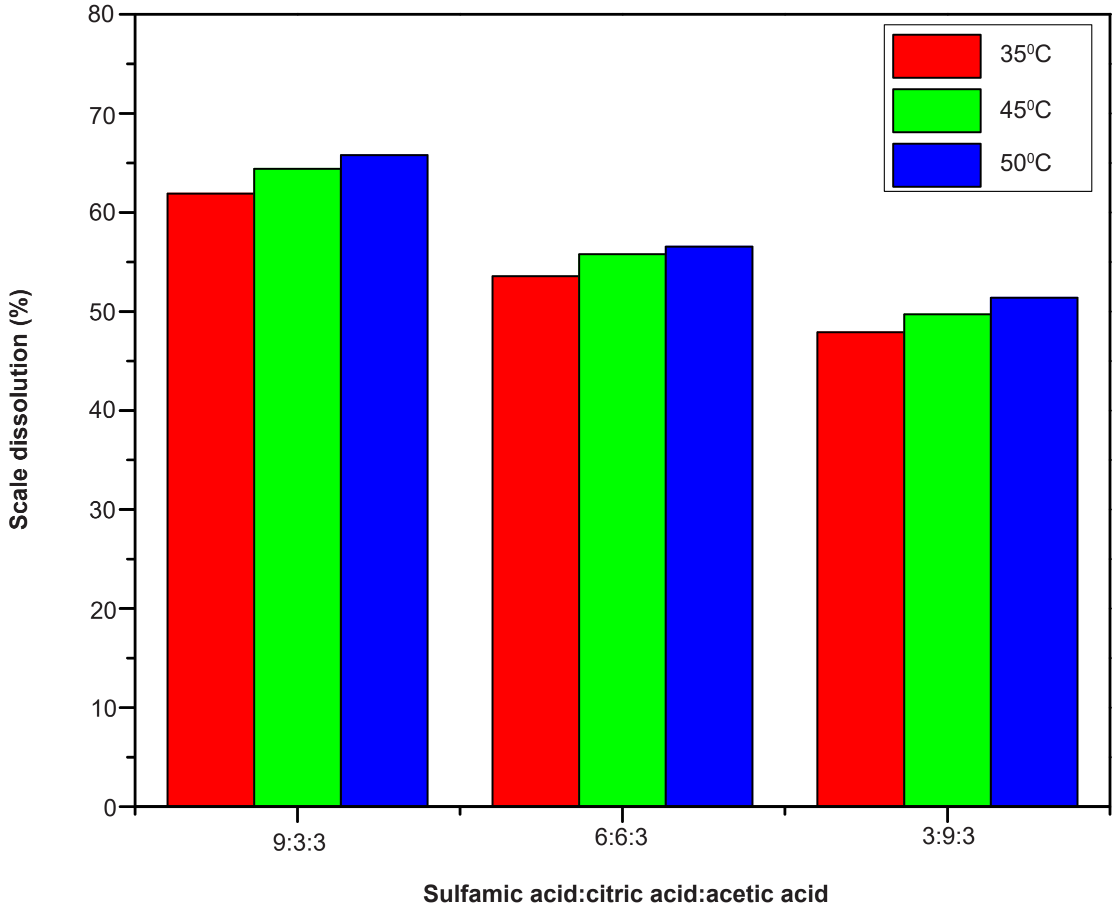

Figure 2. Percentage of dissolved scale based on concentrations of sulfamic acid, citric acid, and acetic acid in the ratio of 9:3:3, 6:3:3, and 3:9:3 on temperature (35, 45, and 50°C) effect.

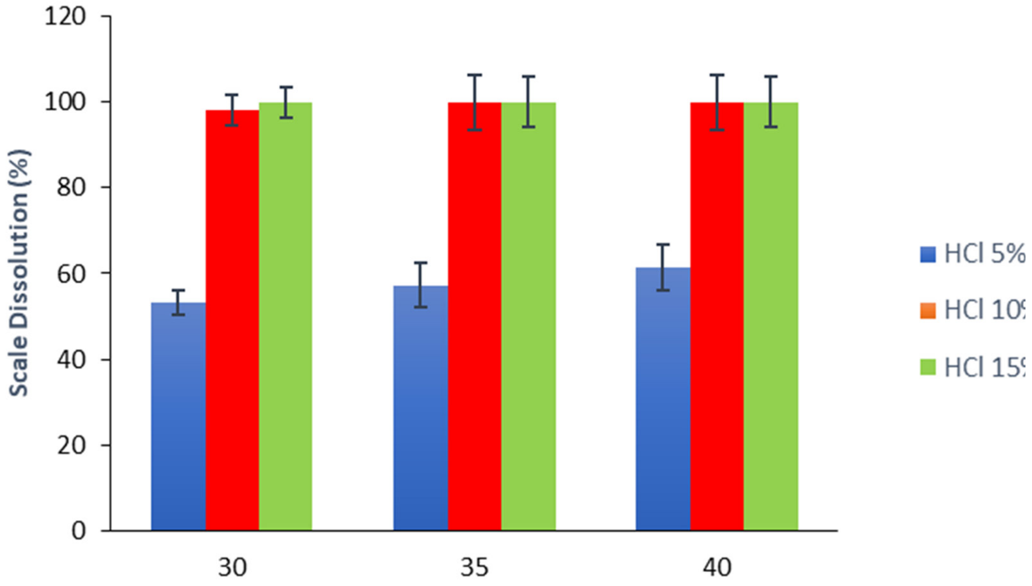

Figure 3. Percentage of scale dissolution based on the effect of HCl concentration and temperature.

effective when using hydrochloric acid solvent. The HCl temperature of 35°C (5%, 10%, and 15%) could dissolve 57.25%, 99.98%, and 100% scale. The increase in scale dissolution with increasing concentration also occurred at test temperatures of 45° and 50°C. The solubility results using hydrochloric acid at a temperature of 45°C were 5, 10, and 15%, respectively, corresponding to solubilities of 61.54, 100, and 100%. Meanwhile, at a temperature of 50°C, the scale dissolver was 63.49, 100, and 100% (5, 10, and 15% of HCl), respectively. The optimum scale dissolver can be achieved with 10% HCl at 45 °C and 50°C temperatures. HCl 15% shows a scale dissolver of 100% at temperatures of 35, 45, and 50°C. This result aligns with previous research, which stated that the higher the acid concentration, the greater the amount of calcium carbonate that can be dissolved. Using 1%, 3%, 5%, 7%, 10%, and 15% of HCl can produce scale dissolution for calcium carbonate, respectively 40%, 58%, 90%, 100%, 100%, and 98% (Murtaza et al. 2022).

The reaction occurs when sulfamic acid, citric acid, acetic acid, and HCl are reacted with calcium carbonate (Equation 4-7) (Guyer, J.P. & PE 2018).

The scale dissolution capability of the combination of the three acids in this study produced the highest value of 65.79% achieved at the ratio of sulfamic acid: citric acid: acetic acid 9:3:3. The scale dissolution value cannot exceed 10% HCl at a temperature of 45°C because HCl is a strong acid that can dissociate perfectly. It gives HCl the ability to dissolve scale vigorously. Previous studies have stated that HCl is better at dissolving scale and can dissolve calcium carbonate of 3500 lb/1000 gal compared to formic acid 700 lb/1000 gal, and acetic acid 400 lb/1000 gal (Mishra et al. 2016). However, the use of HCl can cause corrosion to metal content commonly used in production pipes, and the aggressiveness of the solution changes depending on the concentration, temperature, and presence of oxidizing impurities (such as iron ions, copper ions, and others) (Mishra et al. 2016).

$$CaCO_3(s)+2H_3NSO_3 \rightarrow Ca(SO_3NH_2)_2(aq)\ \text{(calcium sulfamic)}+H_2O(l)+CO_2(g) \quad (5)$$

$$3CaCO_3(s)+2C_6H_8O_7 \rightarrow Ca_3(C_6H_5O_7)_2(aq)\ \text{(calcium citric)}+3H_2O(l)+3CO_2(g) \quad (6)$$

$$CaCO_3(s)+2CH_3COOH \rightarrow Ca(CH_3COO)_2(aq)\ \text{(calcium acetic)}+H_2O(l)+CO_2(g) \quad (7)$$

$$CaCO_3(s)+2HCl \rightarrow CaCl_2(aq)\ \text{(calcium chloride)}+H_2O(l)+CO_2(g) \quad (8)$$

Surface characterization using SEM-EDX

The surface morphology of the $CaCO_3$ scale was studied using SEM. Figure 4 shows the observation results of the $CaCO_3$ scale from the inner layer connected to the steel. As can be seen in Figure 4A, the scale that has been treated with HCl produces a rougher surface compared to Figure 4B, namely the scale soaked in sulfamic acid: citric acid: acetic acid (9:3:3), 4C (6:6:3), and 4D (3:9:3). The SEM results support the results of the gravimetric analysis in Figures 2 and 3.Figures 4C and 4D show the distortion of the scale surface. It can be seen from the many holes on the surface. When sulfamic acid and hydrochloric acid were used in the immersion process, the holes that were formed appeared to disappear.

Table 2 depicts the composition of carbon, oxygen, silicon, and iron for scales immersed with HCl, sulfamic acid, citric acid, and acetic acid (9:3:3, 6:6:3, and 3:9:3). Scale soaked with HCl has the highest Fe content of 51.51%. The Fe composition decreased successively on the scale immersed with sulfamic acid, citric acid, and acetic acid 9:3:3 by 50.16%. The lowest Fe composition can be observed from the scale soaked with a 3:9:3 mixture of sulfamic acid, citric acid, and acetic acid, at 6.71%. These results are inversely proportional to the carbon composition, where the scale soaked with HCl shows the lowest percentage carbon composition of 38.21% compared to the combination of the three acids. The results are in good agreement with the earlier report (Kamal et al. 2018b).

CONCLUSION

The results of this study indicate that a mixture of sulfamic acid, citric acid, and acetic acid with a ratio of 9:3:3 produces the largest percentage scale dissolution (65.79%) compared to the ratios of 6:6:3 and 3:9:3 at a temperature of 50°C. The temperature of the acid mixture, at 35° and 45°C, produces a lower percentage scale dissolution compared to a temperature of 50°C. This result is comparable to 5% HCl at a temperature of 50°C with a scale dissolution of 63.49%. When compared with previous research, the percentage scale removal using organic additives at a temperature of 100°C was 31.91%, indicating that the results of this study are more effective. This

Table 2. The mass percent of scale and acid

Element	HCl	sulfamic acid: citric acid: acetic acid		
		9:3:3	6:6:3	3:9:3
C	38.21±0.24	36.20±0.60	28.95±0.19	62.88±0.24
O	9.42±0.20	10.50±0.49	27.98±0.25	26.50±0.37
Si	0.85±0.07	3.13±0.25	9.47±0.14	1.06±0.06
Fe	51.51±0.72	50.16±1.76	33.60±0.48	6.71±0.26

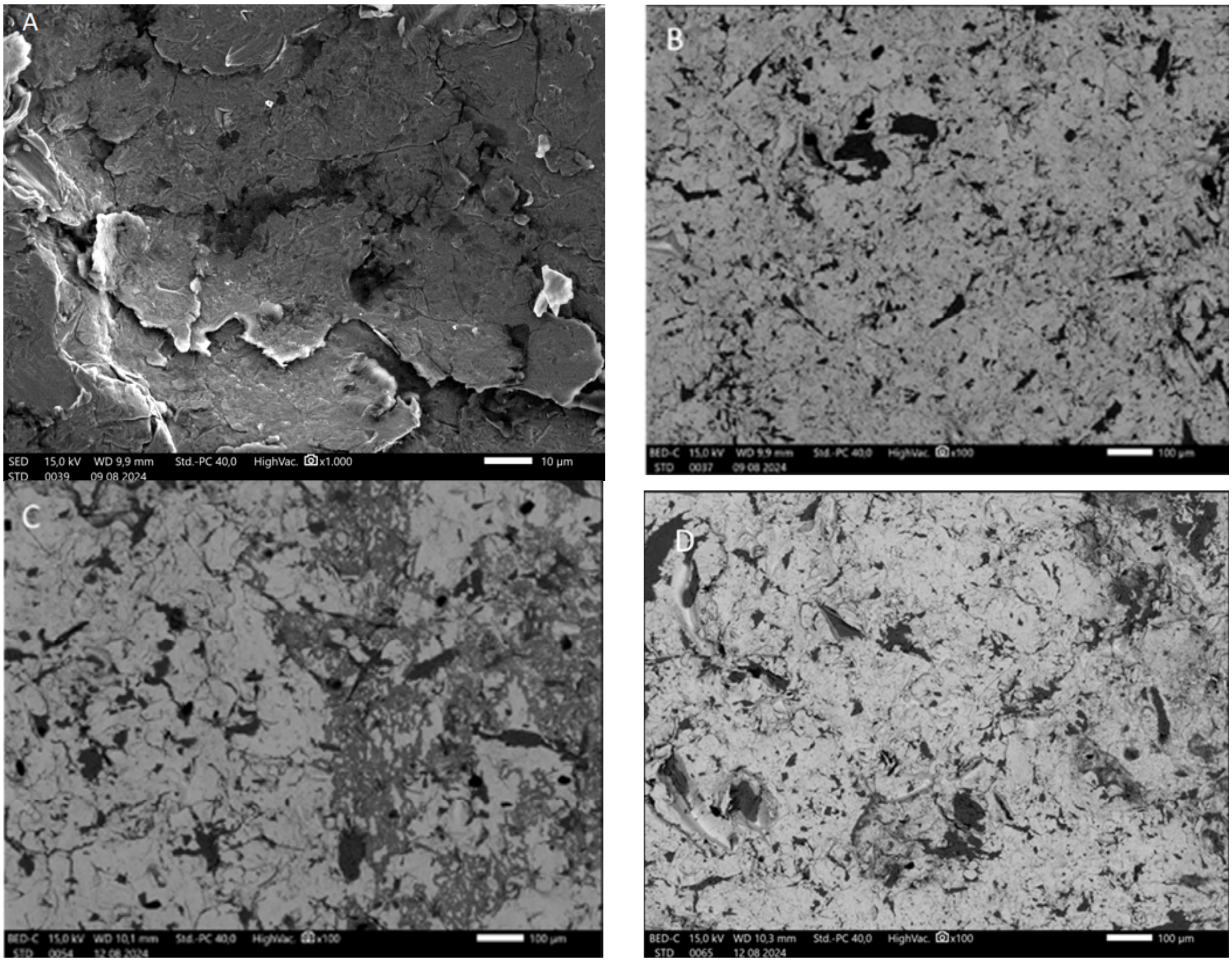

Figure 4. SEM images of the scale inside the pipelines after being immersed in HCl (a), sulfamic acid: citric acid: acetic acid of 9:3:3 (b), 6:6:3 (c), 3:9:3 (d). (Images captured after the scale was immersed in the respective acid at 35°C for 60 minutes).

is because the mixture of sulfamic acid, citric acid, and acetic acid with a ratio of 9:3:3 at a temperature of 50°C produces scale dissolution more than 60%. In brief, a mixture of sulfamic acid, citric acid, and acetic acid with a ratio of 9:3:3 can be used as an alternative scale dissolution at a higher temperature than room temperature with a longer soaking time than HCl. However, the corrosion effect of using the acid mixture can be minimized.

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GLOSSARY OF TERMS

Unit	Definition	Symbol
ASTM	American Standard Testing and Material	
HCl	Hydrochloric acid	
$CaSO_4$	Calcium Sulphate	
$CaCO_3$	Calcium Carbonate	
$BaSO_4$	Barium Sulphate	
ICP-OES	Inductively Coupled Spectroscopy	Plasma-Optical emission
Na_2CO_3	Sodium Carbonate	
SEM-EDX	Scanning Electron Dispersive X-ray Spectoscopy	Microscope-Energy

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