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Optimization of Barium Sulfate ($BaSO_4$) Crystallization Using the Box-Behnken Design Method to Mitigate Scaling in Industrial Pipes

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Abstract

The precipitation of barium sulfate (BaSO4) in industrial piping systems is a major problem because it causes blockage, decreased productivity, and structural damage, including pipe rupture. Although there has been progress in understanding the mechanism of BaSO4 formation, some elements, such as the interaction of tartaric acid with BaSO4 crystals, the best parameters for reducing scaling, and the effectiveness of gas hydrate inhibitors, are still not fully understood. The objectives of this study were to find out how tartaric acid and BaSO4 crystals interact, find the ideal parameters to prevent scale formation, and assess the effectiveness of scaling inhibition techniques using gas hydrate inhibitor and scaling theories. This study used a Box-Behnken experimental design in the laboratory to optimize the temperature, solution concentration, and tartaric acid additive. The test was carried out under laminar flow conditions with a flow rate of 30 mL/min. After that, the morphology and composition of the scale were analyzed by SEM-EDX and XRD. The main results of the study showed that, under ideal conditions, the addition of tartaric acid significantly prolonged the induction time and reduced the mass of BaSO4 scale by up to 50%. This study reached the main conclusion that tartaric acid functions well as an anti-scaling agent and that ideal parameters can be used to effectively prevent scaling. This study is very helpful in developing more efficient and sustainable BaSO4 scaling mitigation methods for industrial applications.

Keywords: *Asam Tartrat, Barium Sulfat ($BaSO_4$), Industrial Piping Systems, Metode Box-Behnken Design, Mitigasi Scaling*

Abstrak

Pengendapan barium sulfat (BaSO4) dalam sistem perpipaan industri merupakan masalah utama karena menyebabkan penyumbatan, penurunan produktivitas, dan kerusakan struktural, termasuk pecahnya pipa. Meskipun ada kemajuan dalam memahami mekanisme pembentukan BaSO4, beberapa elemen, seperti interaksi asam tartra dengan kristal BaSO4, parameter terbaik untuk mengurangi penskalaan, dan efektivitas inhibitor gas hidrat, masih belum sepenuhnya dipahami. Tujuan dari penelitian ini adalah untuk menemukan cara asam tartra dan kristal BaSO4 berinteraksi, menemukan parameter ideal untuk mencegah pembentukan endapan kerak, dan menilai seberapa efektif teknik penghambatan penskalaan menggunakan teori inhibitor dan penskalaan hidrat gas. Penelitian ini menggunakan desain eksperimental Box-Behnken di laboratorium untuk mengoptimalkan suhu, konsentrasi larutan, dan aditif asam tartrat. Pengujian dilakukan dalam kondisi aliran laminar dengan laju aliran 30 mL/menit. Setelah itu, morfologi dan komposisi skala dianalisis dengan SEM-EDX dan XRD. Hasil utama penelitian menunjukkan bahwa, dalam kondisi ideal, penambahan asam tartra secara signifikan memperpanjang waktu induksi dan menurunkan massa skala BaSO4 hingga 50%. Studi ini mencapai kesimpulan utama bahwa asam tartarat berfungsi dengan baik sebagai agen anti-scaling dan bahwa parameter ideal dapat digunakan untuk mencegah kerak secara efektif. Penelitian ini sangat membantu dalam mengembangkan metode mitigasi penskalaan BaSO4 yang lebih efisien dan berkelanjutan untuk aplikasi industri.

Kata kunci: Asam Tartrat, Barium Sulfat ($BaSO_4$), Metode Box-Behnken Design, Mitigasi Scaling, Sistem Perpipaan Industri

1. Introduction

The oil and gas industry faces the problem of barium sulfate ($BaSO_4$) precipitation, often referred to as barite scaling. $BaSO_4$ deposits significantly reduce the permeability of industrial pipes, causing blockages through crystal formation on pore surfaces and throat obstructions [1]. This increases pressure in the pipe and impedes fluid flow, potentially causing structural damage such as pipe rupture [2]. Due to its low solubility and ability to absorb toxic ions such as strontium and radium, operational problems in piping systems are exacerbated [3]. $BaSO_4$ deposits become more difficult to treat under low temperature conditions due to expensive treatment and cleaning. [4].

In oil fields, common minerals, including $BaSO_4$, often cause significant operational problems. The process of producing barium sulfate, or barite, produces scale that reduces the diameter of flow channels and reduces well productivity. Severe equipment erosion is caused by its low solubility, highlighting the importance of preventing its deposition during operations [5]. In offshore oil and gas drilling, the buildup of deposits on oil drilling pipes, such as barium sulfate ($BaSO_4$) deposits, is a significant problem. This can lead to reduced pipe diameter, reduced water transfer, and even the possibility of pipe rupture [6]. Scale can also reduce the diameter of production pipes, inhibit fluid flow, and reduce production volume.

Temperature, ion concentration, and chemical additives are some of the factors that influence $BaSO_4$ crystallization, according to previous research. Using atomic force microscopy (AFM) at 30 °C [7]. The growth behavior and kinetics of barite (001) surface in supersaturated $BaSO_4$ solution were studied. Other studies have shown that large excess of Ba2+ or $SO_4$2− ions inhibits the nucleation and growth of $BaSO_4$, but small excess of Ba2+ ions actually enhances its growth [8]. Because the formation of sulfate minerals such as barite and celestine is ubiquitous in both natural and human-induced systems, the coprecipitation of these minerals has been widely studied. In porous media, coprecipitation causes crystallization of the solid solution, leading to oscillatory zoning, which is rare in bulk solutions or batch experiments [9]. In another study, synthetic $BaSO_4 \cdot CaSO_4$ composites were prepared via coprecipitation technique, characterized, and examined for rare earth element (REE) sorption. The sorption parameters included pH = 4, equilibrium time of 20 min, temperature of 303 K, and a ratio of REE liquid volume to composite mass of 0.2:1 L g-1. [10].

Researchers have also reported the use of additives such as tartaric acid to inhibit crystal growth during scaling processes. Salicylic acid (SA) inhibits the growth of calcium sulfate dihydrate (CSD) crystals with an inhibition efficiency of 98.9% at a concentration of 6 mg/L. This prevents the formation of scale deposits such as carbonate and sulfate in various technological applications [11]. Modified and functionalized polysaccharides serve as effective additives to inhibit crystal growth in scaling processes by interfering with the crystallization mechanism.

Research shows that $BaSO_4$ crystal formation mainly occurs under laminar flow conditions; research shows that longer residence times and greater diffusion penetration lengths result from lower flow rates [12]. Allows for greater deposition near the system inlet [2]. However, experimental studies on the combined impact of flow rate, additive concentration, and temperature on the formation of scaling in the oil and gas industry often use scaling prevention techniques to maintain pipeline efficiency and reduce maintenance costs. To quickly identify scaling problems in oil pipelines, techniques such as gamma ray attenuation and neural networks have been developed [13], Predictive maintenance methods are also used to predict when pigging should be performed, which will reduce maintenance costs and prevent significant production losses [14].

Not much research has been done on the effect of tartaric acid concentration on BaSO4 scaling reduction under laminar flow conditions. Consequently, important questions remain about the mechanism of interaction between tartaric acid and $BaSO_4$ crystals. [15]. Furthermore, there is no relationship between the inhibitory effect of tartrate ions on local corrosion of steel in alkaline solutions and its capacity as a mineral scaling control agent (UK1). Furthermore, the nucleation pathways of calcium sulfate minerals, especially bassanite, from aqueous solutions have been thoroughly studied. However, comparable information regarding $BaSO_4$ is still very limited [16]. The autoxidation of tartaric acid in air-saturated solution with Fe (II) at low pH also exhibits complex chain reaction dynamics. However, no one fully understands its relationship to the crystallization process of $BaSO_4$ [17]. Conversely, it has been shown that the use of tartaric acid as an electrolyte additive in zinc-ion batteries improves performance. However, there has been no report on the use of tartaric acid in controlling $BaSO_4$ scaling [18].

Studies on citric acid as an environmentally friendly additive have shown that it is effective in delaying gypsum nucleation and reducing membrane fouling under certain conditions. However, the ideal parameters for $BaSO_4$ crystallization remain unclear, especially when varying Ba2+ concentrations and temperatures are applied in the piping system [19]. Measurement of the solubility of $BaSO_4$ in water at various temperatures and textures still needs to be done.

In a controlled experimental setting [18]. The interaction between tartaric acid and $BaSO_4$ crystals has not been thoroughly studied in terms of morphology and growth rate. Previous studies have shown that ideal $BaSO_4$ crystallization consists of dispersing 1.19g of $BaSO_4$ in 10 mL of distilled water, adding 11 mL of tetraethyl orthotitanate solution, and heating the mixture. This suggests that Ba2+ adsorption and substrate hydrophobicity are important factors in the organic-water interface crystallization process [20]. With organic additives at various temperatures, understanding of crystal phase evolution and induction time is still limited [21].

In addition, research has not been conducted thoroughly on how the combination of flow rate and additive concentration contributes to scaling prevention in industrial pipes using $BaSO_4$ as a model system [1]. Research on the precipitation of barium sulfate during water flooding processes has been informative, but its application in a broader industrial context is still needed [22]. Scaling theory and gas hydrate inhibition methods have also been developed, but their relevance to $BaSO_4$ scaling remains to be studied [23]. Furthermore, the effects of $BaSO_4$ nanoparticles on the properties of eukaryotic and prokaryotic cells suggest potential biomedical applications. However, their effects on crystallization processes in piping systems remain poorly understood. [17].

Despite significant progress in understanding barium sulfate ($BaSO_4$) precipitation and how to prevent it, several important issues remain unresolved. First, the mechanism of interaction between tartaric acid and $BaSO_4$ crystals under laminar flow conditions has not been thoroughly studied. Consequently, its potential as an anti-scaling agent remains questionable [15]. In addition, the ideal parameters for $BaSO_4$ crystallization are still unknown, especially related to the variation of temperature and Ba2+ ion concentration in the piping system [19]. In addition, systematic research studying the induction time and phase evolution of $BaSO_4$ in the presence of organic additives at various temperatures is still very limited [21]. The combined effect of flow rate and additive concentration in preventing scaling in industrial pipes using BaSO4 as a model system, also to develop an effective strategy in reducing BaSO4 scale in industrial piping systems.

This study is crucial for addressing the significant barium sulfate ($BaSO_4$) scaling that occurs in the oil and gas industry. First, $BaSO_4$ deposits cause pipeline blockages, reduced productivity, and structural damage such as pipe ruptures [1], [2]. This impacts profitability and operations. To reduce maintenance costs, understanding the interaction of tartaric acid and $BaSO_4$ crystals in laminar flow is crucial [15]. Second, the uncertainty about the ideal BaSO4 crystallization parameters, especially related to temperature and Ba2+ ion variations, causes uncertainty in scaling management [19]. Consequently, this problem must be addressed to reduce maintenance costs. Third, there has been no comprehensive research on the benefits of scaling control techniques such as scaling theory and gas hydrate inhibition against $BaSO_4$ [23]. Therefore, the combination of these techniques can produce creative solutions to improve the efficiency of piping systems.

The objectives of this study are to address several gaps in existing knowledge about barium sulfate (BaSO4) precipitation in industrial piping systems. First, to identify the interaction mechanism between tartaric acid and BaSO4 crystals under laminar flow conditions by varying parameters such as tartaric acid concentration, flow rate, and temperature. Second, to determine the ideal parameters to predict conditions for efficiently preventing scale formation. Third, this study will assess the effectiveness of a scaling inhibition method that combines scaling theory and gas hydrate inhibitors. It is hoped that these three objectives will improve the scientific understanding of BaSO4 expansion and develop better mitigation strategies for industrial applications.

2. Material and Method

This study uses a laboratory experimental method to examine the formation of barium sulfate ($BaSO_4$) scale on pipes under laminar flow conditions. The research approach is quantitative, focusing on measuring physical-chemical parameters such as solution concentration, temperature, and the effect of tartaric acid additives on scale formation. The study was conducted in a laboratory using an experimental apparatus specifically designed to simulate laminar flow conditions. To ensure accurate results, this study used a Box-Behnken Design experiment, which allows for the systematic evaluation of combinations of independent variables.

1.1. Research Design

This research design was designed to examine the effect of a combination of independent variables on the formation of barium sulfate ($BaSO_4$) scale under laminar flow conditions. The study used a Box-Behnken experimental design, which allows for a systematic evaluation of three main variables: the concentration of the Ba^{2+} solution (1000 ppm, 2000 ppm, 3000 ppm), the solution temperature (35°C, 40°C, 45°C), and the concentration of tartaric acid additive (0 ppm, 10 ppm, 20 ppm). The dependent variables observed included the mass of scale formed, the conductivity of the solution, and the analysis of the morphology and chemical composition of the scale using instruments such as SEM-EDX and XRD. A total of 15 experiments were conducted based on the experimental design matrix to determine the optimal conditions that can minimize the formation of $BaSO_4$ scale.

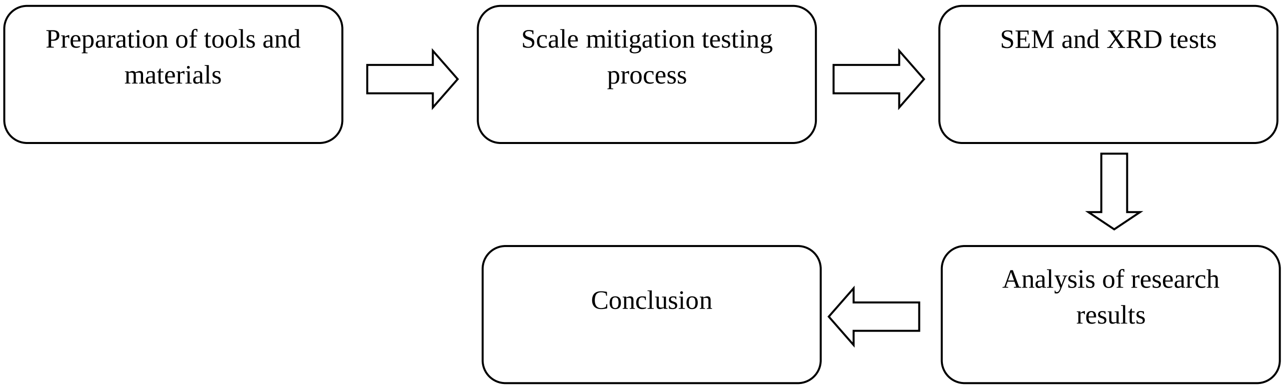

Figure 1. Stages of the research process

1.2. Population and Sample

The materials used in this study consisted of a test solution containing analytical grade $BaCl_2$ (Barium Chloride Dihydrate) crystals, analytical grade Na_2SO_4 (Sodium Sulfate) crystals, distilled water, and tartaric acid ($C_4H_6O_6$) as an additive. The test solution was prepared with varying concentrations of Ba^{2+} solution of 1000 ppm, 2000 ppm, and 3000 ppm, as well as varying concentrations of tartaric acid of 0 ppm, 10 ppm, and 20 ppm. The total volume of solution used for each type of solution ($BaCl_2$ and Na_2SO_4) was 2.5 liters, with each solution flowing through a laminar flow system to a stainless steel coupon as a medium for depositing $BaSO_4$ scale. The resulting scale samples were then collected from the coupon surface after the experimental process for 2 hours. A total of 15 samples were analyzed.

1.3. Instruments

The instruments used in this study include laboratory equipment specifically designed to support barium sulfate ($BaSO_4$) scale formation experiments under laminar flow conditions. The main equipment includes a water pump to regulate the solution flow rate of 30 mL/min, a conductivity meter to measure the solution conductivity in real-time during the experiment, and a heater with a temperature sensor to control the solution temperature at 35°C, 40°C, or 45°C. In addition, a stainless steel coupon was used as a scale deposition medium, while an analytical balance was used to measure the mass of the formed scale. For further analysis, instruments such as SEM-EDX (Scanning Electron Microscopy-Energy Dispersive X-ray) were used to study the morphology and chemical composition of the scale, and XRD (X-Ray Diffraction) to identify the crystal phase of the $BaSO_4$ scale.

1.4. Research Procedures

The research procedure was carried out systematically in several stages to ensure the data obtained were accurate and representative. The study began with the preparation of the test solution, namely dissolving $BaCl_2$ and Na_2SO_4 crystals in distilled water with varying Ba^{2+} concentrations of 1000 ppm, 2000 ppm, and 3000 ppm, and adding tartaric acid as an additive at concentrations of 0 ppm, 10 ppm, and 20 ppm. The solution was then flowed through a laminar flow system using a water pump with a flow rate of 30 mL/min, while the solution temperature was kept constant at 35°C, 40°C, or 45°C using a heater and temperature sensor. During the experimental process, the conductivity of the solution was measured periodically using a conductivity meter, with measurement intervals of every 2 minutes in the first hour, 6 minutes in the second hour, and 15 minutes in the third hour. After 2 hours of experiment, the stainless steel coupons were removed, dried in an oven at 80°C for 1 hour, and weighed to determine the mass of the crust formed. The formed crust was then analyzed using SEM-EDX to study the morphology and chemical composition, and XRD to identify its crystalline phase. A total of 15 experiments were conducted according to the Box-Behnken experimental design to ensure the test covered all combinations of independent variables.

3. Results and Discussion

The addition of tartaric acid significantly reduced the mass of barium sulfate scale, as shown in Table 1, as a result of optimization using the Box-Behnken Design method. The combination of parameters generated by this optimization can be used to effectively prevent scaling.

Table 1. Results of research optimization using Box-Behnken Design.

Run No	**Concentration (ppm)**	**Temperature (^0C)**	**Tartaric Acid Additive (ppm)**	**Scall Mass (gram)**
	X1	**X2**	**X3**	**Y**
1	1000	35	10	0.0897
2	3000	35	10	0.1628
3	1000	45	10	0.0728
4	3000	45	10	0.1525
5	1000	40	0	0.1226

Run No	Concentration (ppm)	Temperature (^0C)	Tartaric Acid Additive (ppm)	Scall Mass (gram)
	X1	X2	X3	Y
6	3000	40	0	0.2136
7	1000	40	20	0.0714
8	3000	40	20	0.1426
9	2000	35	0	0.1385
10	2000	45	0	0.1226
11	2000	35	20	0.1128
12	2000	45	20	0.1096
13	2000	40	10	0.1182
14	2000	40	10	0.1182
15	2000	40	10	0.1182

The amount of crust formed was positively correlated with the concentration of the Ba2+ solution. Conversely, the addition of tartaric acid inhibited crystal growth and significantly reduced the mass of the crust (Figure 2). In addition, the mass of the crust increased as a result of increasing temperature due to the acceleration of the chemical reaction of crystal formation.

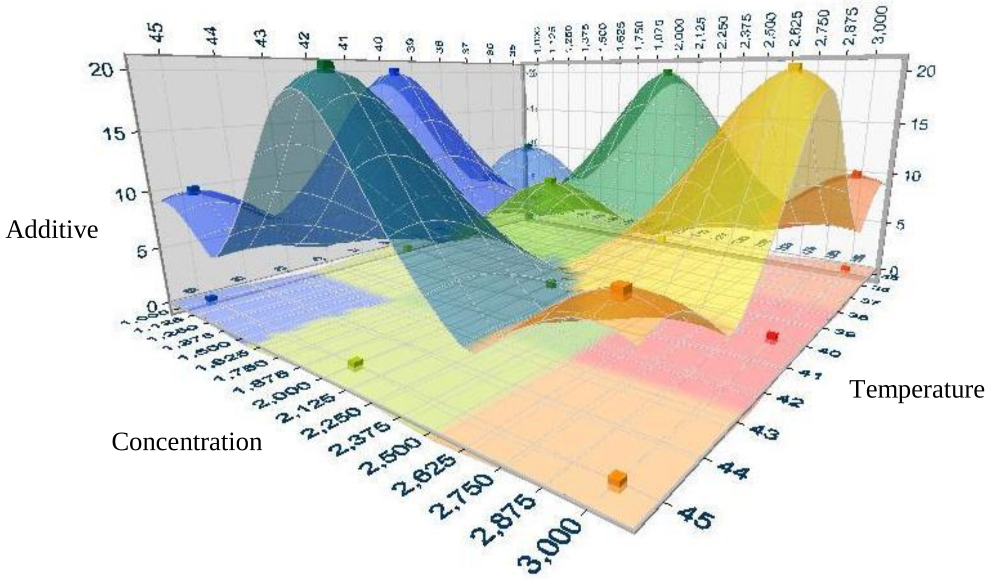

Figure 2. The relationship between the mass of barium sulfate crust and variations in solution concentration (1000, 2000, 3000 ppm Ba^{2+}), temperature (35, 40, 45°C), and tartaric acid (0, 10, 20 ppm).

By adding tartaric acid, the average growth rate of barium sulfate crystals is slower. This occurs because a higher concentration of tartaric acid reduces the crystal growth rate, resulting in a smaller crystal mass (Figure 3).

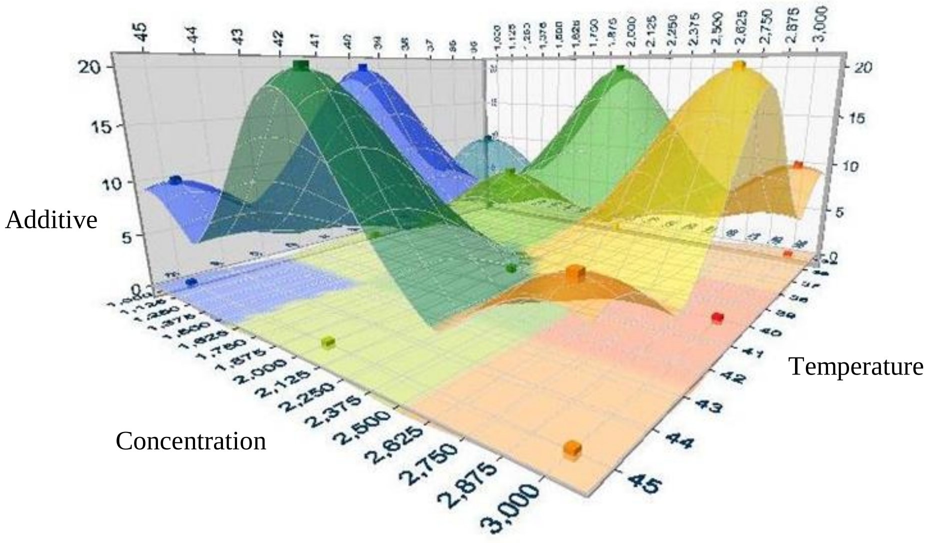

Figure 3. Average growth rate of barium sulfate crystals at varying solution concentration, temperature, and tartaric acid additives.

Figure 4 shows that barium sulfate crystals without additives have a pointed shape that tends to stick to the pipe surface, but the addition of tartaric acid makes the crystal morphology flatter, which makes it difficult to stick to the pipe surface. This shows the ability of tartaric acid to stop the formation of scale which can be seen in figures 5 and 6. The more additives added, the more rounded the barium sulfate crystals that occur, meaning that the barium sulfate crystals will dissolve easily so that the process of forming barium sulfate crystals is less in the pipe.

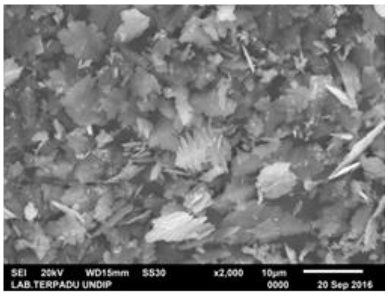

Figure 4. Crystal morphology of barium sulfate without tartaric acid additive.

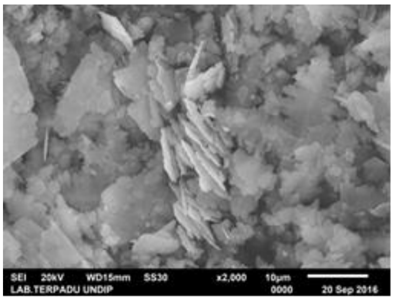

Figure 5. Morphology of barium sulfate crystals with 10 ppm tartaric acid additive.

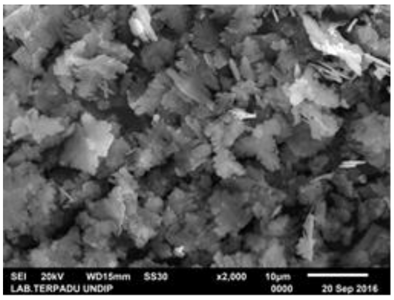

Figure 6. Morphology of barium sulfate crystals with 20 ppm tartaric acid additive.

The interaction mechanism between tartaric acid and BaSO4 crystals under laminar flow conditions. Figures 1 and 2 show experimental results indicating that the addition of tartaric acid significantly prolongs the induction time of BaSO4 crystal formation. Its ability to inhibit crystal nucleation through adsorption on the surface of growing crystals demonstrates the ability of tartaric acid as an anti-scaling agent. Previous studies have found that organic additives such as tartaric acid can prevent the nucleation and development of sulfate minerals by stopping the reaction process of scale-forming ions [24]. In addition, Su et al. (2024), stated that the use of organic additives can reduce the crystal growth rate by changing the crystal morphology [25].

Physicochemical factors such as temperature, solution concentration, and tartaric acid additive significantly influence the formation of barium sulfate ($BaSO_4$) scale. Experimental results (Table 1) show that higher Ba2+ solution concentrations result in greater scale mass, while the addition of tartaric acid additives significantly reduces the scale mass. Due to the increased kinetic energy of the ions in the solution, increasing the temperature also accelerates scale formation. These results are in line with research by Parafiniuk & Siuda, (2021), which found that the growth and nucleation process of sulfate mineral crystals can be accelerated by high temperatures [26]. Besides that Kumar et al. (2018), stated that the formation of scale minerals such as calcium carbonate can be controlled by optimizing physical-chemical parameters such as ion concentration and temperature [27].

The results of this study indicate that the presence of organic additives such as tartaric acid affects the induction time of barium sulfate ($BaSO_4$) crystal formation at various temperatures. This demonstrates the ability of tartaric acid to inhibit crystal nucleation and growth by adsorbing onto the surface of developing crystal nuclei. This result is in line with the work Al-Gailani et al.(2024), where researchers found that organic additives have the ability to slow down the formation of calcium carbonate crystal nuclei through an adsorption mechanism [28]. Sarrigiannidis et al. (2020), found that organic additives such as polymers or surfactants can change the morphology of crystals and reduce their growth rate [29].

The combination of a low flow rate (30 mL/min) and the addition of a tartaric acid additive reduced the formation of barium sulfate ($BaSO_4$) scale in industrial piping systems. Although low flow rates can increase the residence time of the solution in the pipe, which can accelerate scale formation, experimental results show that the addition of tartaric acid significantly slows this process (Figure 2). Even under laminar flow conditions with low flow rates, tartaric acid can be used to reduce scaling. This result is in line with research Al-Gailani et al.(2024), who found that scale mineral formation can be reduced by using organic additives and controlling physical parameters such as flow rate. Freitas et al.(2024), shows that optimizing additive concentration can improve scaling prevention efficiency under various flow conditions [30].

The addition of tartaric acid significantly altered the morphology of barium sulfate (BaSO4) crystals. BaSO4 crystals without the additive were pointed and tended to adhere strongly to the pipe surface, as shown by SEM analysis (Figures 3, 4, and 5). In contrast, the crystal morphology became flatter with the addition of tartaric acid, which made the crystals more easily carried away by the flow and less likely to adhere to the pipe surface. This suggests that tartaric acid inhibits crystal nucleation and alters the crystal structure to prevent scaling. This result is in line with research Freitas et al. (2024), where researchers discovered that organic additives have the ability to change the crystal form of minerals such as calcium carbonate, making them more stable and less aggressive in forming deposits. Feigelson (1996), found that changing crystal morphology through the adsorption of organic additives can reduce the ability of crystals to adhere to material surfaces. This finding supports these findings. Therefore, this study provides new information on how crystal morphology changes can be used to reduce scaling in industrial piping systems [31].

4. Conclusion

The process of barium sulfate deposition in the test tube is determined by measuring the conductivity of the solution exiting the test tube. The solution's conductivity and the test duration are used to determine the induction time. The induction time is the time required for the ions in the solution to react to form the first crystal nucleus. The concentration of the solution, temperature and addition of tartaric acid additives affect the induction time and growth rate of the barium sulfate crystals formed. The concentration of the solution, temperature and addition of tartaric acid additives affect the morphology and chemical composition of the barium sulfate crystals formed, but do not affect the crystal phase of the barium sulfate formed because there is only one crystal phase of barium sulfate, namely barite.

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