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Treatment of Silicate Ion with *Bacillus subtilis* Bacteria in Demineralization-Water/Steam Cycles in Power Plants

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Abstract

Inorganic silica and various mineral deposits are particularly important in the process waters of power plants. The presence of these inorganic species, especially silica, poses many challenges for process water applications in power plants. If silica in process waters is not controlled, it forms hard, difficult, and dangerous deposits in the process water. Silica formation and accumulation cannot be prevented by many conventional methods and scale inhibitors. In this study, *Bacillus subtilis* bacteria were used to minimize silica formation in the process water of power plants. For this purpose, many different parameters were optimized in the system steps. Under the optimum experimental conditions, the detection limit for SiO_2 was determined as 0.0375 mg/mL, the quantitation limit as 0.1136 mg/mL, and the correlation coefficient as 0.9997. Additionally, recovery values range between 96.5% and 103.2%, indicating the accuracy of the method. The results obtained are promising for the use of silica removal in process water applications. In addition, the use of *Bacillus subtilis* bacteria for the treatment of process water will provide significant economic benefits. Therefore, this study will make an important contribution to the literature and will be very advantageous in terms of cost for various industrial organizations that face silica problems in process waters.

Keywords: SiO_2, *Bacillus subtilis*, energy, water treatment, demineralization

1. Introduction

An ideal boiler water treatment system consists of a reverse osmosis unit, thermal degasser, pressure filter, and softening unit to ensure the flow of high-purity feed water. If normal hard tap water is used as additional water in steam boilers, silica begins to accumulate on the heating surface of the boiler after a while. This consumes more energy and requires the use of more chemicals and large amounts of acid to clean the inside of the boiler. Although the water softening in the system helps to prevent scaling, after a while, it becomes insufficient, and more systemic problems arise. This is undesirable in terms of both time and cost [1,2]. Good pre-treatment, which is the building block of every system, is very important in terms of providing high standards of feed water to the system [3]. A schematic representation of feed water pretreatment for boilers is given in Fig. 1.

Silica and mineral deposits can become serious operational problems for untreated or poorly treated process waters [4,5]. Poorly soluble electrolytes found in process water include calcium oxalate, silica, strontium sulfate, barium, carbonate, phosphate, calcium, and others. The constituents of these electrolytes vary depending on many variables such as pH, temperature, water chemistry, conductivity, etc. Among these mineral deposits, magnesium silicate and silica are particularly problematic. Since they cause excessive amounts of sediment formation, they cause operational failures that cause catastrophic damage in process water systems [6]. In industrial water systems, especially silica causes serious problems that cannot be solved. Uncontrolled silica will accumulate in significant quantities throughout the process, resulting in the formation of hard and challenging deposits that are difficult to clean. Conventional cleaners cannot counteract the silica build-up and are also dangerous as they try to get rid of the silica by chemical treatment. It will also cause significant corrosion over time. This will reveal another problem that silica will create [5].

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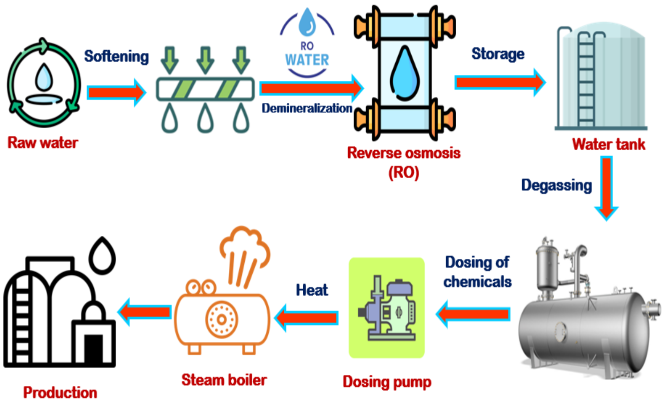

Figure 1. Schematic representation of feedwater pretreatment for boilers

Silica deposits accumulating in equipment such as distillation units, engine jackets, boilers, cooling tower fillings, steam generators, heat exchangers, evaporators, etc. cause inefficient heat transfer and a significant decrease in heat transfer rates. Failure in one or more of the successive steps will greatly affect the other steps. One of them is that organic and inorganic deposits in paper and pulp mills cause significant losses in production. Another is the accumulation in boiler pipes in industrial water systems. A visual of the accumulation of silica and mineral deposits is shown in Fig. 2 [7]. This buildup in boiler tubes reduces equipment efficiency, leading to increased fuel consumption and overheating in the system. In parallel, the accumulation of poorly soluble silica in the system creates a complex matrix environment. This will directly affect corrosion exposure, creating negative consequences for the system [8].

Figure 2. Silica and mineral deposits

Many methods have been proposed to improve the quality of process water and, furthermore, to ensure that wastewater is purified and reused. In addition, with the advancement of technology, many different systems have been developed and used [9–11]. These include biofloc technology, recirculating aquaculture systems, and aquaponics [12,14]. These methods are generally expensive, can cause contamination by introducing different pathogens into the system, and are laborious. Other disadvantages include the fact that they often cause confusion in systems and also pose a threat to human health through accumulation. For this reason, the method that has been used more popularly in recent times to improve wastewater quality and provide treatment of process water is to use *Bacillus subtilis* bacteria. The method using *Bacillus subtilis*, which also has probiotic properties, is quite remarkable in that it is effective against a wide range of pathogens and has more advantages than others in improving the quality of process water [15,16]. In this study, experiments are presented to see what kind of change occurs when *Bacillus subtilis* bacteria are applied to all steps of the system to improve process water quality and reduce silica accumulation.

2. Materials and methods

2.1. Chemicals and reagents

All chemicals used in this study were of analytical grade and used without further purification. Molybdic acid (MoO_3-$(H_2O)_2$), hydrochloric acid (HCl), sodium hydroxide (NaOH), oxalic acid ($H_2C_2O_4$), and 4-(methylamino)phenol sulfate were used to form the reducing solution, and sodium disulfite chemicals were obtained from Merck. Silica stock solutions at a concentration of 100 mg mL^{-1} were prepared with sodium metasilicate (Na_2SiO_3) in deionized water. Working solutions of silica at different concentrations were prepared by diluting the main stock solution to appropriate concentrations. *Bacillus subtilis*, the biosorbent used in this study, was isolated from thermal water deposits in the lower region of Pamukkale travertines in Türkiye. 250 mL of Nutrient Broth (NB) medium was used to grow *Bacillus subtilis* bacteria. The pH of the medium was adjusted with 0.1 mol/L HCl or NaOH to ensure fermentation and then autoclaved. After the autoclaving step, 2.5 mL (3.6×10^{7}) of cell suspension was inoculated into each glass vial. Then, it was shaken at 120 rpm at 35 ^{o}C.

2.2. Instruments

The Water Story brand Dream Plus I Finesta model deionized water device was used to prepare the solutions required for the experimental studies. The Shin

Saeng brand SHPM-10 model magnetic stirrer was used to adjust the sample temperature. The WTW brand Inolab Multi 9630 IDS model pH meter was used to measure the instantaneous pH of the samples taken from each step of the demineralization system. Weighing of the chemical materials used to prepare the solutions during the study was carried out using the Mettler Toledo brand ML204T model precision balance. The measurements of the samples taken from each step of the water-vapor cycle were performed on a WTW brand AE-S60-2U model spectrophotometer. Specific conductivity measurements of the samples taken in the water-steam cycle were carried out on the WTW brand Cond 3110 model device. The determination of silica was carried out at a wavelength of 800 nm.

2.3. Procedure

This method was based on the measurement of biosorption of silica on *Bacillus subtilis*. To investigate the effectiveness of bacteria on silica accumulated in the system, a 50 mL water sample taken from the process was placed in a polypropylene volumetric flask. Before the addition of *Bacillus subtilis*, the process water sample had a total silica concentration of 41.8 mg/L, with 37.6 mg/L in dissolved form and 4.2 mg/L as suspended silica. Other physical and chemical characteristics of the raw water sample were as follows: pH 7.3, electrical conductivity 1560 µS/cm, total dissolved solids (TDS) 1648 mg/L, total hardness 90.1 ºFr, calcium 185.8 mg/L, magnesium 106.1 mg/L, bicarbonate 413 mg/L as $CaCO_3$, sulfate 550 mg/L, and total organic carbon (TOC) 5.7 mg/L. Then, 3 mL volume of *Bacillus subtilis* suspension (corresponding to a concentration of 3×10^8 CFU/mL) was applied to determine the amount of silica. Silica analysis was performed according to the Advancing Standards Transforming Markets (ASTM) standards [17,18]. First of all, a sample with a 3 mL concentration of *Bacillus subtilis* suspension was taken, and optimum conditions for the process were pH 8.7 and room temperature. And the preliminary step required for silica determination was completed. Then, a 10 mL volume of this sample with optimum conditions was taken into a polypropylene volumetric flask, and 2 mL of 10% (w/v) molybdic acid solution was added. Then, this mixture was stirred in a magnetic stirrer for 10 minutes. After this step, a 2 mL volume of 4-(methylamino)phenol sulfate ($C_{14}H_{20}N_2O_6S$) reducing solution was added to the volumetric flask, and the stirring process was continued for another 10 minutes. After the reaction, the sample prepared for measurement was transferred to the UV cuvette, and analysis was performed using the UV-Vis spectrophotometer. All experiments were performed in three replicates. A schematic representation of the procedure in the study is given in Fig. 3.

3. Results and discussion

3.1. Effect of waiting time on *Bacillus subtilis* activity

Samples taken from each step of the demineralization and boiler water systems to which *Bacillus subtilis* was added were kept for 0-72 hours, and the change in SiO_2 content was examined. The procedure in the study was applied to samples taken from each step of the system, and when the values obtained as a result of the analysis were examined, it was determined that very small decreases occurred in the amount of silica with *Bacillus subtilis* bacteria up to 48 hours of waiting and that silica removal was fixed after 48 hours. This result showed that the *Bacillus subtilis* activity and removal were still at high levels up to 48 hours to perform silica analysis in water samples taken from all steps of the process. When the same procedure was applied to water samples taken from the process steps at the end of the 48 hours, the analysis result showed that the activity of *Bacillus subtilis* decreased. What is intended to be explained in this section is that the conditioning with *Bacillus subtilis*

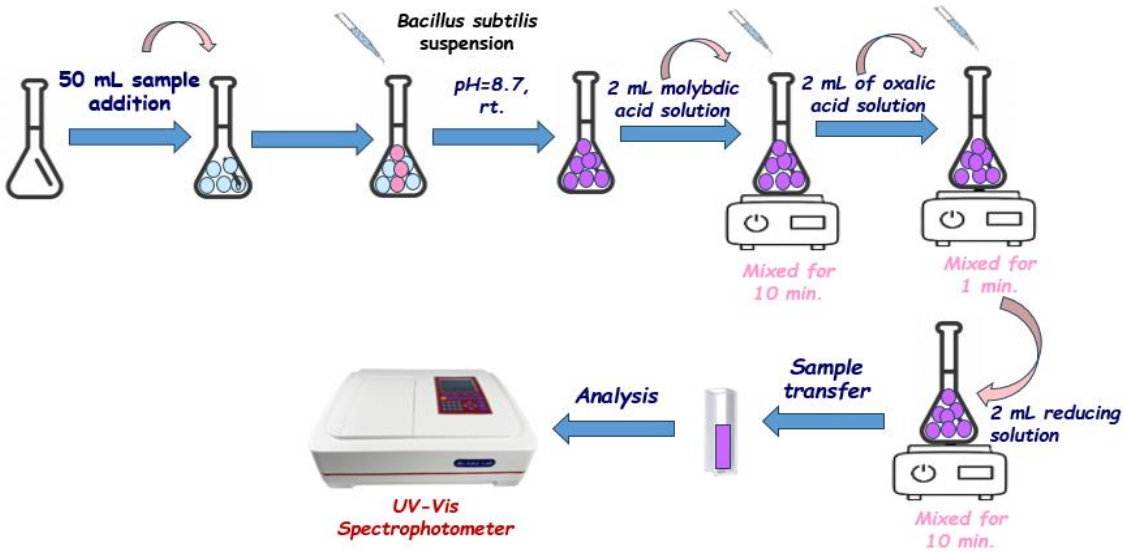

Figure 3. Schematic representation of the procedure

bacteria can be applied immediately without waiting for any period of time in water samples taken from process steps. The procedure was applied to determine the direction of its effectiveness both as soon as the sample is taken from the process steps and when it is incubated with *Bacillus subtilis* for the specified periods. As a result, the optimization made in the 0-72 hour range is to check whether the effectiveness of *Bacillus subtilis* bacteria continues after how many hours the sample is left. It was shown that this value was fixed at the end of the 48 hours, and the removal value decreased. The effect of waiting time on *Bacillus subtilis* activity is shown in Fig. 4.

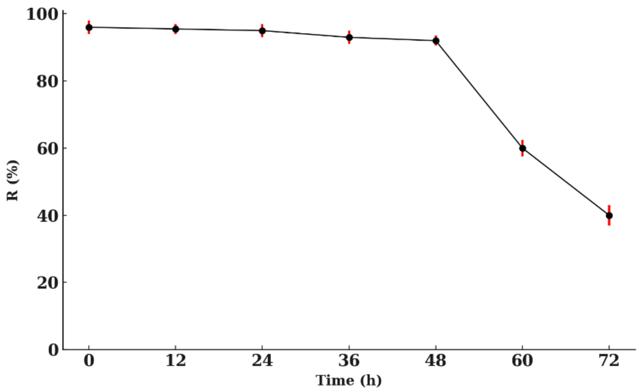

Figure 4. Effect of waiting time on *Bacillus subtilis* activity

3.2. Effect of pH on *Bacillus subtilis* activity

The aim of pH optimization is to control the system's operation in the ideal pH range when the *Bacillus subtilis* suspension is added. The pH change was controlled at different stages of the process, and optimization was carried out at certain pH ranges to determine the required value for all stages of the process. According to the results obtained from all stages of the process, pH control provides a more reliable assessment of bacterial activity under changing conditions. For this purpose, a procedure was applied between pH 5-11 values to determine the effect of pH on silica removal in all stages of the process. The optimum pH value at which silica removal was achieved for all process steps was determined as 8.7 [19,20]. The effect of pH on *Bacillus subtilis* activity is shown in Fig. 5.

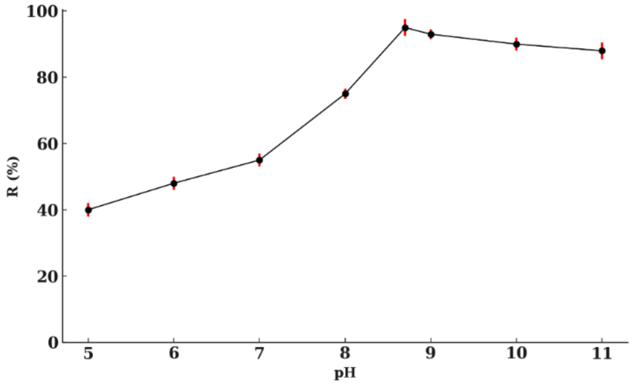

Figure 5. Effect of pH on *Bacillus subtilis* activity

3.3. Demineralization system

3.3.1. Effect of Bacillus subtilis on silica concentration in raw water

In the study, the specific environmental conditions of the sample to be analyzed for raw water when it is first taken are important. Therefore, the initial temperature, pH, and conductivity values at the point where the raw water sample was taken were measured. It was determined that the initial temperature at the point where the raw water sample was taken was 15 °C, the pH was 7.23, and the conductivity was 1726 µS/cm.

The values obtained after applying the procedure to perform SiO_2 analysis in raw water are given in Fig. 6. When Fig. 6 is examined, the effect of mixing time on the effect of *Bacillus subtilis* bacteria in raw water is examined. As a result of the examinations, it was seen that there was an increase in removal up to 10 minutes, but there was no difference in removal after 10 minutes. For this reason, it was seen that 10 minutes of mixing time was sufficient for the analysis in raw water.

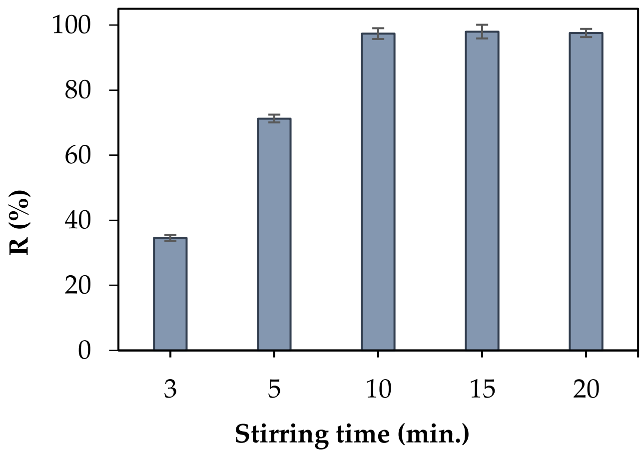

Figure 6. Effect of mixing time on removal in raw water.

3.3.2. Effect of Bacillus subtilis on the pretreatment plant

The pre-treatment process takes feed water from raw water. The ideal operating temperature of the pre-treatment and subsequent demineralization process is 15-35 °C. In addition, the ideal operating temperature varies seasonally between 15-35 °C. For this reason, *Bacillus subtilis* activity was examined at these temperatures, and its effect was evaluated (Fig. 7). The activity of *Bacillus subtilis* was examined by pre-conditioning with *Bacillus subtilis* in the mentioned temperature range. It was observed that there was very little change in the amount of SiO_2 as the temperature increased. It was also found that *Bacillus subtilis* bacteria worked effectively at 30 °C.

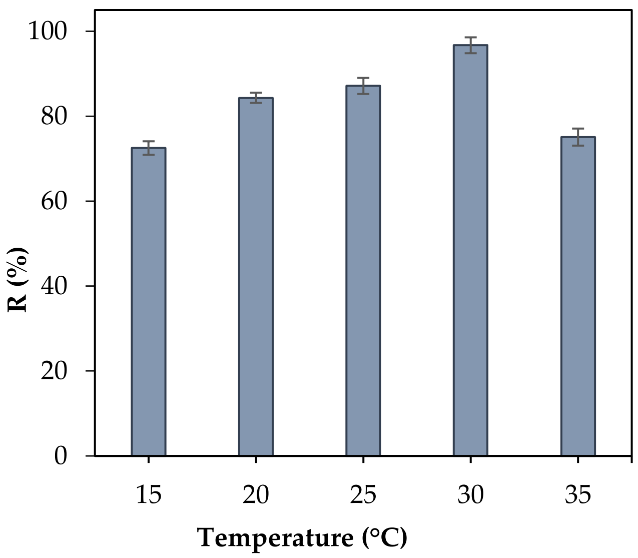

Figure 7. Effect of temperature on removal in the pretreatment plant.

3.3.3. Effect of Bacillus subtilis on reverse osmosis system

The formation of insoluble metal silicates in reverse osmosis is another problem. To prevent this situation, the softening process must be carried out appropriately. Therefore, samples taken from the feed water were examined in detail in the laboratory. First of all, specific environmental conditions in the field were determined to determine the conditioning rates in the process. Then, the initial temperature, pH, and conductivity values at the points where the sample was taken before reverse osmosis were measured. From the on-site measurement values of the water sample before reverse osmosis, it was found that the temperature was 20 °C, the pH was 7.20, and the conductivity was 1510 µS/cm. Among the chemicals dosed before reverse osmosis, silica-based antiscalant was used for membrane protection, and NaOH and HCl were used to balance the pH of the system. The effect of *Bacillus subtilis* on SiO_2 in the presence of these chemicals used in the treatment before reverse osmosis was also investigated. The process was carried out by adding 0-10 mL of *Bacillus subtilis* bacteria to the raw water for analysis. As a result of SiO_2 analysis, while the amount of SiO_2 in the raw water sample without *Bacillus subtilis* bacteria was quite high, it was found that the SiO_2 removal in the raw water sample with 3 mL of *Bacillus subtilis* bacteria was high. It was determined that the value remained constant when more was added. For this reason, the study was continued with the optimum value of 3 mL of bacteria.

3.3.4. Effect of Bacillus subtilis on reverse osmosis membrane and effluent SiO_2 concentration

Biofilm growth in reverse osmosis membranes significantly reduces the water velocity inside the membrane. For this reason, when microbiological organisms enter the membrane, they reproduce here and form a biofilm. As a result, the pressure loss in the membranes increases, the production flow rate decreases, and as a result, the production efficiency of the system decreases, and causing production losses. However, instead of the antiscalant given before the membrane, the solution created with the addition of 5-10-15-20-30% *Bacillus subtilis* bacteria for about one month was dosed into the system to condition the system. Determination of these dosage rates was preferred to be the same as the dosage rate of the existing antiscalant chemical to facilitate performance-cost comparison. Membrane efficiency was kept under observation during the one-month trial period. It has been determined that high efficiency is achieved in removing the amount of silica when the same amount of *Bacillus subtilis* bacteria is used instead of the existing antiscalant chemical. This led to the conclusion that the bacteria used are beneficial to the system, do not form a biofilm, reduce the amount of flux passing through the membranes, increase the membrane permeability, and are effective in the treatment of other impurities in the water, such as SiO_2. High-pressure pumps are used for the effective operation of the reverse osmosis system. The operating pressure of the sample plant is 16-32 bar max, 50 Hz. The analysis results of the study showed that *Bacillus subtilis* was not affected by the operating pressure in this range. No additional chemical treatment was applied during the study period.

3.3.5. Effect of Bacillus subtilis on electro-deionization system efficiency and product water SiO_2 concentration

Pretreatment with *Bacillus subtilis* increased the permeability of the Electro-Deionization (EDI) system and improved the water quality of the system. In the study conducted on the EDI product water according to the procedure, it was observed that positive improvements were experienced as the amount of wastewater decreased and the product capacity increased. In addition, this process maintained the stability of ideal pH (8.7) and pressure conditions (2 bar). It was determined that it did not affect the pH, which is one of the important parameters for the process of the system in this study, during the flow, and stabilized the system operating pressure. In addition, *Bacillus subtilis* conditioning was performed before EDI, and from the analysis results, it was seen that it provided an improvement equal to the amount of chemical used in the system, corresponding to 30%. In addition, the increase in membrane permeability of the EDI module is understood from the stability of pH and pressure, the stability of wastewater pressure, wastewater amount, and the stability of product water amount (maximum product water amount determined by the EDI manufacturer = 10 tons). It has also been observed that membrane life can be extended and used beyond 2 years when *Bacillus subtilis* conditioning is applied instead of chemical conditioning.

3.4. Boiler system

3.4.1. Effect of Bacillus subtilis on boiler feed water

Since silicates form highly persistent deposits in boiler tubes, their volatile carryover to turbine components is a critical issue. The composition of boiler feedwater must allow impurities to concentrate within reasonable levels without exceeding the tolerance limits of the specific boiler design. If the feedwater does not meet these requirements, pretreatment is necessary to remove impurities. However, complete removal of impurities is not always required, as chemical treatment within the boiler can effectively and economically eliminate them. The quantity and nature of impurities affect system purity, with certain impurities, particularly silica, being of greater concern. The impurity requirements for any given feedwater depend on the feedwater usage rate and the specific boiler design (pressure, heat transfer rate, etc.). Consequently, feedwater purity requirements can vary significantly [16].

Boiler water softening chemicals are added continuously or intermittently depending on the hardness of the water and other factors, and the chemicals added to react with dissolved oxygen and the chemicals used to prevent scale formation and corrosion in the water supply system are continuously supplied to the water supply system during the process. A sample was taken from the boiler for analysis at 45 ^{o}C. It was left to cool in the cooler, and the sample was processed after the temperature was brought to normal. This situation represents the desired situation for the boiler system. According to the ABMA (American Boiler Manufacturers Association) standard, the system operates under ideal conditions. 0-30 mL of *Bacillus subtilis* bacteria was added to the boiler feed water. This value was determined according to the chemical ratio used in boiler feed water conditioning. Maximum silica removal was obtained as a result of using 20 mL of bacteria. The obtained result is given in Fig. 8.

3.4.2. Importance of conditioning for turbine

Direct steam conditioning is not applied under any circumstances, and therefore, the chemical quality of the steam depends on the measures applied to control the feedwater and boiler water. Therefore, feedwater and boiler water conditioning are very important. Because one of the most important objectives is to prevent deposits and corrosion in the steam path, for example, in pipes, valves, and turbine parts. The purity of the steam must be high. Silica is the most soluble of the common boiler water contaminants in high-pressure steam and has a high volatility. It can become supersaturated during expansion in the turbine. Its deposits on the blades cause loss of turbine efficiency and serious damage. Salts deposited in the steam pipe under load cause the development of concentrated solutions on unloading, following the entry of significant air or condensation of residual steam (Fig. 9) [21]. This effect is a situation that should be taken into consideration, especially for reheaters, turbines, and some feed heaters. In addition to causing mechanical failure, the stress occurring during turbine operation can initiate other forms of corrosion, such as corrosion cracking.

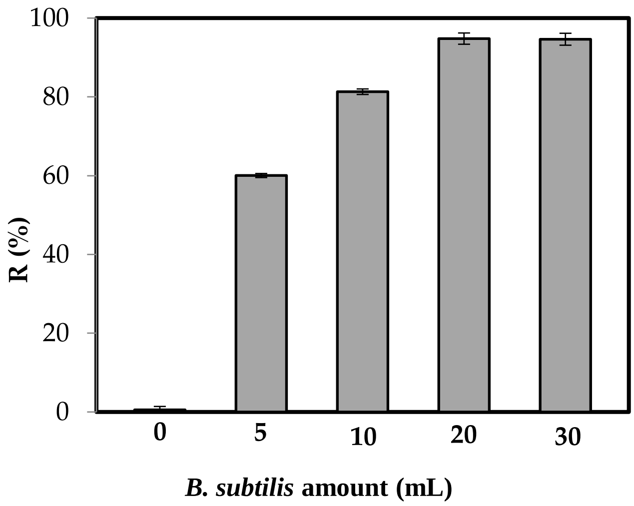

Figure 8. Effect of *Bacillus subtilis* on boiler feed water

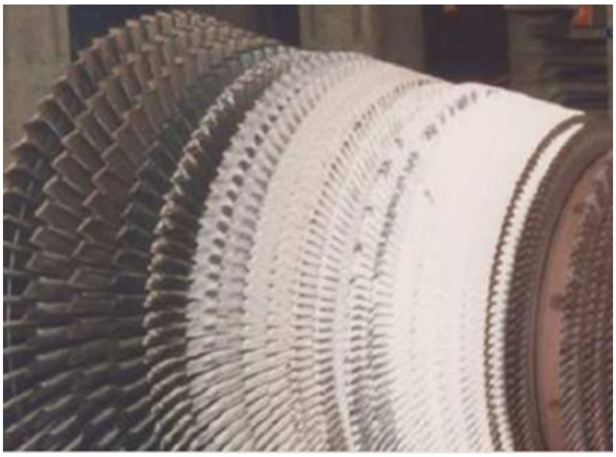

Figure 9. Damage caused by silica on turbine blades.

During the periodic maintenance after conditioning, it was observed that the turbine blades were not fouled and were clean after long-term use without much need for a suitable stabilizer. This indicated that the steam carried in the system was adequately conditioned in the feed and boiler water and that the *Bacillus subtilis* conditioning also reduced silica and other impurities in the feed water.

3.5. Method validation

Some validation parameters, such as the calibration equation, detection limit, recovery values, correlation coefficient, and quantitation limit of the method used for the analysis of SiO_2, were investigated. All analyses were performed at 25°C. Each solution was prepared in triplicate to ensure calibration accuracy and validate the analytical method. A calibration graph was created by evaluating the data obtained as a result of the analysis in the study. To determine repeatability, intraday and interday standard deviations were examined. The relative standard deviation (%RSD) value between the outcomes of five distinct sample solutions generated at the same concentration was less than 2%, indicating the analytical method's precision. In this study, the lowest analyte concentration (LOD) and the lowest analyte concentration values that can be determined with acceptable repeatability and accuracy (LOQ) were determined using the values of 3Sb/m and 10Sb/m. Where Sb is the standard deviation and m is the slope of the calibration equation. Under the optimum experimental conditions, the detection limit for SiO_2 was determined as 0.0375 mg/mL, the quantitation limit as 0.1136 mg/mL, and the correlation coefficient as 0.9997. Additionally, the calibration equation is $y = 0.0253x + 0.0032$, and the recovery values vary between 96.5% and 103.2%, demonstrating the accuracy of the method.

4. Conclusion

Chemical pollution in water, air, and soil is becoming more and more alarming day by day. This study aims to replace chemical treatment in power plants or all processes that require a water/steam cycle with an economical and environmentally beneficial one. For this reason, some preliminary research has been carried out on the silica problem, one of the biggest problems in closed cycles. While the field application of this study was carried out, preliminary tests were carried out to see what effect it has on the removal of silicate-induced pollution in water and steam cycles in industry. To investigate the effect of the parameters taken from different steps, such as temperature and mixing time in the water and steam cycles of a private industrial establishment, and which are more important for each step, *Bacillus subtilis* bacteria were added to samples containing silica, and the results obtained were examined. The biosorption of silica on the *Bacillus subtilis* bacterium largely depends on the active sites of the biomass. Since they have both a live and active metabolism, resistance and biosorption mechanisms act as a whole. Biosorption of silica was achieved effectively thanks to the active sites of the *Bacillus subtilis* bacterium. When the results obtained in the study were analyzed, a more environmentally friendly way was followed by using *Bacillus subtilis* bacteria instead of toxic chemicals to reduce the amount of silica. In addition, one of the important results of the study is that *Bacillus subtilis* has been found to reduce not only silica but also the amount of impurities in the water. In addition to silica, this study revealed that *Bacillus subtilis* treatment effectively reduced other waterborne impurities. Specifically, in raw process water and boiler systems, notable reductions were observed in total hardness (from 1.76 to 0.88 mg/L), magnesium (from 106.1 to 54.8 mg/L), calcium (from 185.8 to 91.2 mg/L), and total dissolved solids (TDS). These reductions were achieved without the need for conventional chemical dosing, highlighting the dual benefit of this biological approach in both silica and secondary impurity control. At the same time, it creates a preliminary idea by providing a different dimension for many similar studies. Most importantly, by strongly affecting the needs in the field of wastewater treatment, added value will be provided on behalf of our country, especially in the economic sense, on an issue that exists in the industry and is a constant problem. In light of the information obtained as a result of this study, the fact that it will both protect the environment from tons of chemicals used for the steam water cycle and help to provide a great economic gain reveals the other advantageous aspects of the study. It will also be beneficial to extend the life of the process and provide ease of operation.

Conflict of interest

The authors declare that they have no conflict of interest.

Author contributions

Deniz Bozkurt: Material preparation, Formal analysis, Investigation, Writing–original draft.

Aslıhan Dalmaz: Data curation, Writing–original draft, Writing–review & editing, Conceptualization.

Sefa Durmuş: Conceptualization, Methodology, Writing–review & editing, Funding acquisition, Supervision.

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