

OPTIMIZATION OF DESCALING CONDITIONS OF THERAPY POOLS IN TOURIST HEALTH CENTER "BANJA KULAŠI"

ORIGINAL SCIENTIFIC ARTICLE

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ABSTRACT:

One of the problems in spa centres with thermal water is the deposition of scale on the overflow of therapeutic pools. In practice, scale is removed by washing with a solution of technical hydrochloric acid at a concentration of 10–16 w/w%. However, this process causes damage to the grout and adhesive, resulting in tile detachment. This study aimed to investigate the possibility of removing scale without significant damage, based on the chemical composition of thermal water, scale, and grout samples taken from the Health and Tourism Centre "Banja Kulaši." Chemical analysis of the scale confirmed that it is of carbonate type (94.32% CaCO₃). The solubility of the scale and grout was tested in two ways – by immersion and by spraying with HCl solutions of varying concentrations. The immersion method showed a total solubility of the examined scale (>98%), while the spraying method aimed to simulate real cleaning conditions of therapeutic pools with different contact times. The solubility of the scale using the spraying method was also high (>92%) for contact times of 5, 10, and 15 minutes. The solubility of the grout depends on both the concentration of HCl and the contact time.

KEYWORDS: thermo-mineral water, descaling, scale solubility, grout solubility

INTRODUCTION

Thermal and spa tourism, as well as the use of thermal, mineral, and thermomineral water, is becoming increasingly popular, not only for health services but also for health protection and relaxation. Bosnia and Herzegovina is rich in various mineral, thermal, and thermomineral waters; there are 193 deposits of mineral water, 57 of thermal water, and 30 of thermomineral water, with around 400 natural springs and 170 boreholes and wells [1]. The thermal spring near Prnjavor, the Banja Kulaši health and tourism complex, has water with a specific chemical composition, high pH value, and temperature above the average for natural spring water, which places it in the group of hypothermal waters [2]. Due to its specific composition, low dissolved salt content, and high temperature, this water is suitable for treating skin diseases such as atopic dermatitis and is also beneficial for other therapeutic applications [3].

One of the issues in spa centres with thermal water is the buildup of scale on the overflow edges of therapeutic pools. Performing treatments or altering the chemical composition of the water to maintain

hygiene conditions is undesirable, as this changes the composition, specific properties, and potential health benefits of the water [4].

The removal of scale is done by washing with technical hydrochloric acid at an approximate concentration of 10 w/w%, which effectively dissolves the accumulated scale. Along with scale removal, components from the grout that binds the tiles also dissolve, causing damage to the tile structure and resulting in tiles detaching from the edges of the pool. Therefore, it is essential to know the exact chemical composition of the thermal water used in health-tourism and spa centres, as well as the nature of the scale deposits, to ensure proper maintenance and cleaning of the pools while adhering to all hygiene measures and standards.

This study aimed to propose the most efficient method for maintaining and cleaning the pools in the health-tourism centre "Banja Kulaši" to prevent issues with the pool's appearance and preserve the characteristics and therapeutic properties of the water used in the pools. An effort was made to propose the most effective cleaning agent for the pools, based on

the precise chemical composition of the water, scale, and grout used for tile adhesion. At the same time, the descaling agent should not be overly aggressive to avoid damaging the pool tiles. During the experimental work, in addition to laboratory analyses to determine the chemical composition of the water, scale, and grout, the effectiveness of scale and grout dissolution was tested. The solubility testing was carried out in two ways – by the immersion method and by the spraying method using HCl solutions of varying concentrations.

EXPERIMENTAL

For this research, the spa water from the Health-Tourism Centre "Banja Kulaši" was analysed, along with the scale deposits that form as the spa water cools down, and the grout used to fill the spaces between the tiles in the spa pool.

Sampling and analysis of thermal water

The thermal water used for analysis was sampled at the Health-Tourism Centre "Banja Kulaši".

Table 1. Tested water quality parameters and standard testing methods used

Tested parameter	Standard method
pH	Electrochemical
Electrical conductivity	Conductometry
Temperature	Thermometer
Alkalinity	Titrimetric with HCl
Water hardness	Complexometric titration
Cl ⁻	Argentometric titration
Oxidizability	Titration according to Kubel-Tiemann
Dissolved oxygen	The Winkler method
SO ₄ ²⁻	Indirect iodometric titration
Dry residue	Gravimetric
NH ₄ ⁺	Spectrophotometric with Nessler's reagent (without distillation)
NO ₂ ⁻	Spectrophotometric with sulfanilic acid
NO ₃ ⁻	UV spectrophotometric
Orto phosphates	Spectrophotometric with ammonium molybdate and ascorbic acid
Fe	Spectrophotometric with 1,10-phenanthroline
SiO ₂	Spectrophotometric with ammonium molybdate
Mn	Spectrophotometric via permanganate
Al	Spectrophotometric with eriochrome cyanine R

A clean 3 dm³ plastic canister, rinsed with the water being sampled, was used for sampling. Water quality parameters were determined according to the Standard Methods for the Examination of Water Quality [5] as shown in Table 1. All tested water quality parameters were performed in triplicates.

Sampling, preparation, and analysis of scale and grout

Sampling of the scale was conducted in the drainage pit of the thermal spa water. The first step in preparing the sample for chemical analysis was its grinding in a ball mill. The ground sample was then sieved through a 0.3 mm mesh and then dried in an oven at 105 °C to a constant weight. The chemical analysis of the scale was performed using standard gravimetric analysis for testing limestone [6].

The grout used for the analysis was prepared according to the manufacturer's instructions. After curing, the grout was ground, sieved, and dried for chemical analysis, following the previously mentioned standard procedure [6].

Study of the solubility of scale and grout

The study of the solubility of scale was conducted in two ways.

The first method involved immersing the unground scale in solutions of different concentrations of mineral (HCl) and organic (citric) acids. The solubility of scale in mineral acid was tested at different concentrations of HCl (3.0 w/w %, 5 w/w%, and 10 w/w%), while the solubility of scale in citric acid was tested at acid concentrations of 5 w/w%, 10 w/w%, and 20 w/w%.

The experiment was conducted as follows:

1. Appropriate solutions of HCl and citric acid were prepared with distilled water.
2. Weighing dishes with filter paper (white strip) were dried in an oven at 105 °C, cooled, and weighed.
3. Samples of unground scale (~1 g) were weighed into 3 beakers, and a solution of acid ($V=100\text{ cm}^3$) of different concentrations was added to each beaker. The contact time between the scale sample and the acid was 5 minutes (simulating real time cleaning conditions from Banja Kulaši).
4. The separation of the solid and liquid phases was performed by filtration through previously dried and weighed filter paper (white strip).
5. The filter paper and the residual content on it were dried in an oven at 105 °C, cooled, and weighed.

To achieve a more complete simulation of the cleaning conditions of the pools used in the Health Tourism Centre "Banja Kulaši", a second method for determining the solubility of scale was applied—spraying the scale with solutions of HCl of different concentrations and varying contact times between the acid and the scale. The pool cleaning simulation was conducted as follows:

1. To form a layer of deposited scale in a porcelain dish (previously dried and weighed), daily evaporation of 100 cm³ of thermal spa water was performed in a water bath for 7 days. The porcelain dish was then dried in an oven at 105 °C, cooled, and weighed to determine the mass of the formed scale.
2. After the formation of the scale layer, the scale was sprayed with 5 cm³ of 5 w/w% HCl using a sprayer, at different contact times (5 min, 10 min, 15 min).
3. The porcelain dishes with scales were washed with distilled water, then dried in an oven at 105 °C, cooled, and weighed.

The study of the solubility of the grout was conducted using both the immersion method and the spraying method. The immersion method was performed in the same way as for the solubility of scale, while the solubility testing by spraying was conducted by spraying the unground sample of grout on filter paper, using different contact times.

Based on mass difference before and after treatment with acids, the efficiency of dissolving scale by immersion and spraying, as well as the efficiency of dissolving the grout, solubility and removal efficiency, was calculated using the following formula:

$$\text{Removal efficiency (\%)} = \frac{m_0 - m_1}{m_0} \dots \dots \dots (1)$$

where: m_0 is the mass of the weighed sample of scale or grout (g) or the mass of the formed scale in the porcelain dish, m_1 is the mass of the sample of scale or grout remaining on the filter paper after filtration or in the remaining mass of scale in porcelain dish (g).

RESULTS AND DISCUSSION

The obtained results and discussion of the conducted research on thermal water, scale, and grout, as well as the solubility of scale and grout, are divided into sections.

Results of the chemical analysis of thermal water

The results of the conducted chemical analysis of the thermal water from "Banja Kulaši" are presented in Table 2.

Table 2. The results of the chemical analysis of thermal water from "Banja Kulaši"

Tested parameter	Results	
Temperature (at the spring source) (°C)	28	
pH (at 19.2 °C)	11.85	
Electrical conductivity (at 19.2 °C) (µS/cm)	642.0	
Dry residue (at 180 °C) (mg/dm ³)	197±0.00	
Alkalinity (mg CaCO ₃ /dm ³)	p-alkalinity	132.6±1.26
	m-alkalinity	163.8±1.53
Water hardness (mg CaCO ₃ /dm ³)	Total	101.0±0.00
	Carbonate	101.0±0.00
	Non-carbonate	0.00
Ca ²⁺ (mg/dm ³)	39.64±0.00	
Mg ²⁺ (mg/dm ³)	0.48±0.00	
Fe (mg/dm ³)	<0.10	
Mn (mg/dm ³)	<0.025	
Al (mg/dm ³)	<0.06	
SiO ₂ (mg/dm ³)	5.95	
Consumption of KMnO ₄ (mg KMnO ₄ /dm ³)	1.05±0.10	
Oxidizability (mg O ₂ /dm ³)	0.27±0.10	
Dissolved oxygen (at 26 °C) (mg/dm ³)	3.12±0.03	
OH ⁻ (mg/dm ³)	17.23±0.01	
CO ₃ ²⁻ (mg/dm ³)	37.35±0.60	
Cl ⁻ (mg/dm ³)	23.55±0.16	
SO ₄ ²⁻ (mg/dm ³)	3.82±0.07	
NH ₄ ⁺ (mg/dm ³)	0.18	
NO ₂ ⁻ (mg/dm ³)	<0.001	
NO ₃ ⁻ (mg/dm ³)	<0.10	
Orto phosphates (mg PO ₄ ³⁻ /dm ³)	<0.05	

Based on the results presented in Table 2, it can be concluded that this is soft water, as the total hardness of the water was found to be 101.0 mg CaCO₃/dm³ [7]. The tested water is a specific case of groundwater, as groundwater generally has a significantly higher content of mineral salts and, consequently, higher water hardness [8]. According to the classification of natural mineral waters based on the value of dry residue determined at 180 °C, as prescribed by European legislation (Directive 2009/54/EC), the water from Banja Kulaši falls into the category of

waters with low mineral content (range of values 50-500 mg/dm³) since it has a dry residue value of 197 mg/dm³ [4]. The analysis of carbonate hardness yielded a value greater than the value of total water hardness, which represents a specific case in water analysis. This relationship between carbonate and total hardness indicates that, in addition to Ca²⁺ and Mg²⁺ ions, carbonates and hydroxides of alkali metals, primarily sodium or potassium, are present in the water, which are part of alkalinity but not of water hardness. The label on the bottled water from Banja Kulaši indicates the presence of sodium ions at a concentration of 29.00 mg/dm³ and potassium ions at a concentration of 1.00 mg/dm³. The low salt content in the tested water is also confirmed by the low value of the dry residue mentioned earlier.

From the ration between *p* and *m* alkalinity, the presence of hydroxide ions (OH⁻) was established at a concentration of 17.23 mg/dm³ and carbonate ions (CO₃²⁻) at a concentration of 37.35 mg/dm³. The presence of these types of ions (OH⁻ and CO₃²⁻) directly indicates that bicarbonate ions (HCO₃⁻), which are the most prevalent in natural waters, are not present in the water. Therefore, this ionic composition makes the thermal water of Banja Kulaši specific. Additionally, due to the presence of OH⁻ and CO₃²⁻ ions, which cannot be present in a neutral or slightly alkaline environment, the pH of the tested water is also specific. The pH of the thermal water is 11.85, classifying it as highly alkaline water [9]. Generally, groundwater has a pH in the neutral or slightly acidic range due to the presence of dissolved gases (primarily CO₂) [10].

The characteristic of groundwater is often the high content of iron and manganese [8], [10]. The conducted analysis of tested water sample found a concentration of Fe less than 0.10 mg/dm³ and a concentration of Mn less than 0.025 mg/dm³.

The analysis of anions revealed a significant presence of chlorides, at a concentration of 23.55 mg/dm³, and sulfates, at a concentration of 3.74 mg/dm³.

Based on the presented results, it can be concluded that the thermal water from Banja Kulaši is water with a low amount of organic matter and nutrients, and it has a distinctly specific composition, both in terms of pH value and the content of ions present in it.

Results of the chemical analysis of scale and grout

The results of the chemical analysis of the scale formed by cooling of the thermal water and the grout used to fill the gaps between the tiles of the spa pool are presented in Table 3.

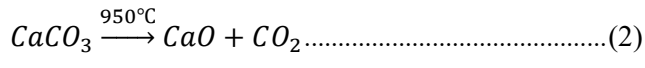
Table 3. The results of chemical analysis of scale and grout

Tested parameter	Results (w/w%)	
	Scale	Grout
Loss on ignition	43.95±0.04	33.26±0.12
Moisture	1.54±0.10	4.43±0.02
Insoluble residue	0.50±0.04	6.24±0.155
SiO ₂	1.06±0.01	16.11±0.10
Fe ₂ O ₃	0.12±0.00	0.12±0.01
Al ₂ O ₃	0.03±0.02	1.01±0.15
CaO	52.82±0.16	39.59±0.11
MgO	0.55±0.04	4.33±0.09
Sulfates	0.09 ± 0.02	0.69±0.05

Based on the results presented in Table 3, it is evident that the dominant component in the tested scale sample is CaO.

The formation of scale is influenced by multiple factors, and the chemical analysis of the water identified quality parameters that create ideal conditions for the precipitation of CaCO₃. Since the thermal water is highly alkaline (with a measured pH of 11.85), contains Ca²⁺ and CO₃²⁻ ions, and has a temperature of 28 °C, the cooling of the water, particularly at the overflow edge of the pool, leads to the formation of scale [11].

Loss on ignition is one of the parameters of chemical analysis that can determine the composition of scale. During the calcium carbonate calcination, the following reaction occurs:



Theoretical mass loss of pure CaCO₃, according to the mentioned reaction, is 44% [12], while the experimentally obtained value for loss on ignition was 43.95%, which is very close to the theoretical value. The content of CaCO₃ in tested scale sample was calculated from the CaO content using the formula:

$$CaCO_3(\%) = CaO(\%) \cdot \frac{M(CaCO_3)}{M(CaO)} \dots\dots\dots (3)$$

The obtained content of CaCO₃ in the scale sample of 94.32% classifies the tested scale into the group of carbonate scales. The fact that the content of CaCO₃ in the scale is not entirely pure indicates the possible presence of silicate components found in the thermal water.

Similarly, the content of CaCO₃ in the grout sample was calculated to be 70.70%, which is also confirmed by the high value of loss on ignition (33.26%). The significant presence of CaCO₃ in the grout is the main reason for its dissolution during the washing of therapeutic pools with technical HCl.

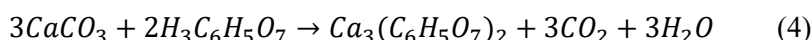
However, a considerably higher concentration of SiO_2 (16.11%) was found in the grout, indicating that minerals from cement clinker, an indispensable inorganic mortar binder, are present in the grout [13]. This could be confirmed by mineralogical analysis.

Results of the solubility of scale and grout

Immersion method

The results of the solubility testing of scale with different concentrations of citric acid using the immersion method are presented in Table 4.

The dissolution of carbonate scale in citric acid is represented by the reaction:



However, the obtained tricalcium citrate is insoluble in water [14], so the efficiency of dissolving CaCO_3 is low at low concentrations of citric acid in a neutral medium. In an alkaline environment ($\text{pH} > 13$), there is a significant increase in the solubility of tricalcium citrate [15]. Using 5 w/w% citric acid, the efficiency of dissolving CaCO_3 was 67.81% (Table 4), and increasing the concentration of citric acid to 10 w/w% did not result in a significant increase in the solubility of CaCO_3 . According to literature [16], only with the formation of mono- and di-calcium citrates does solubility significantly increase, but much higher concentrations of citrate ions are required for their formation. An increase in solubility (76.00%) was observed when using 20 w/w% citric acid. However, increasing the concentration of citric acid is not cost-effective for use under real conditions, and satisfactory efficiency in removing scale was not achieved. Therefore, the investigation of the solubility of scale with citric acid was no longer a subject of this research.

Fig. 1 shows the efficiency of dissolving scale and grout using the immersion method in different concentrations of HCl, with a contact time of 5 minutes.

Table 4. Efficiency of dissolving scale using the immersion method in citric acid with a contact time of 5 min

Citric acid concentration (w/w%)	Dissolving efficiency (%)
5	67.81
10	67.42
20	76.00

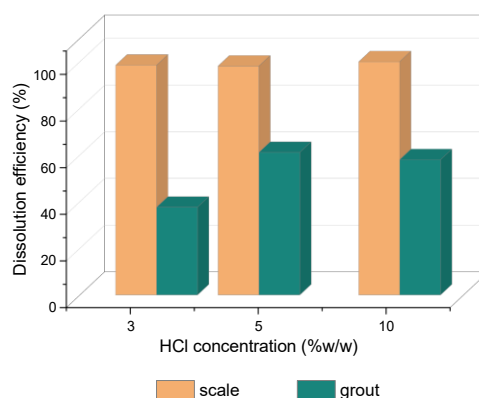


Figure 1. Efficiency of dissolving scale and grout using the immersion method in HCl solution of different concentrations with a contact time of 5 min

Fig. 1 shows that the obtained efficiency of scale removal was very high and consistent for all the applied concentrations: 98.13% for 3 w/w% HCl, 98.15% for 5 w/w% HCl, and 99.4% for 10 w/w% HCl. However, the solubility of the grout increases with the increase in HCl concentration. The mass loss of grout with 3 w/w% HCl application was much lower (37.64%) compared to the loss with 5 w/w% or 10 w/w% HCl (58.2% and 58.6%, respectively). Cleaning the therapeutic pools in Banja Kulaši is carried out using technical acid with a concentration of 10 w/w% HCl, and based on the presented results, it is clear why there is frequent separation of tiles at the edge of the pool (Fig. 2).



Figure 2. Appearance of damaged pool tiles caused by cleaning with technical HCl (10 w/w%)

The maximum solubility of scale and grout was obtained using the immersion method with a contact time of 5 minutes. Given that the difference in the solubility results obtained using 5 w/w% HCl and 10 w/w% HCl was minimal, concentrations of 3 w/w% HCl and 5 w/w% HCl were used for further testing.

Spraying method

In real conditions, in practice, cleaning of therapeutic pools is not performed using the immersion method, but rather through spraying with acid. Therefore, to simulate real conditions, the spraying method was also conducted. A comparative presentation of the solubility of scale and grout using the spraying method with 3 w/w% HCl and 5 w/w% HCl at a contact time of 5 minutes is shown in Fig. 3.

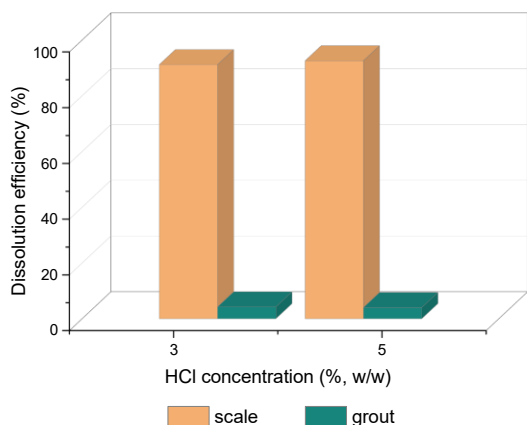


Figure 3. Solubility of scale and grout using the spraying method with 3 w/w% HCl and 5 w/w% HCl at a contact time of 5 min

The efficiency of dissolving scale and grout using the spraying method does not differ much between the applications of 3 w/w% and 5 w/w% HCl (Fig. 3). The efficiency of scale dissolution using the acid is high, exceeding 90% for both tested concentrations, while the solubility of the grout is significantly lower (around 4%) in both cases.

To determine the dependence of dissolution on contact time, the formed scale and grout were dissolved using the spraying method with 5 cm³ of 5 w/w% HCl at different contact times (Fig. 4).

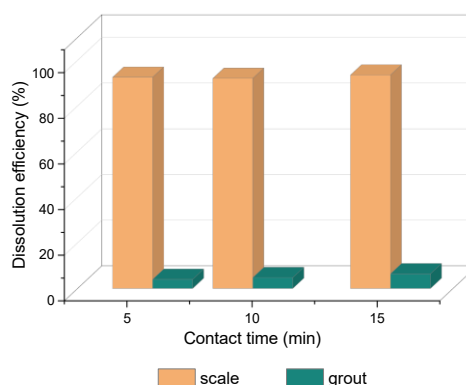


Figure 4. Dissolution of scale and grout using the spraying method with 5 cm³ of 5 w/w% HCl at different contact times

Fig. 4 shows that the efficiency of scale dissolution using the spraying method is very high and satisfactory, similar to the results obtained with the immersion method. Using a 5 w/w% HCl solution, the dissolution efficiency of the scale was 92.57% at a contact time of 5 minutes, 92.06% at 10 minutes, and 93.53% at 15 minutes. Testing the solubility of the scale using either the immersion or spraying method resulted in nearly identical efficiency (>90%), with neither the acid concentration nor the contact time having a significant influence.

However, the dissolution efficiency of the grout using the spraying method with 5 w/w% HCl (Fig. 4) shows considerably lower values compared to the results obtained by the immersion method with 5 w/w% HCl (58.2%) (Fig.1). The solubility of the grout using the spraying method with 5 w/w% HCl was 4.25% at a contact time of 5 minutes, 4.78% at 10 minutes, and 6.42% at 15 minutes.

A comparative graphical representation of the mass loss of grout for both tested methods is shown in Fig. 5.

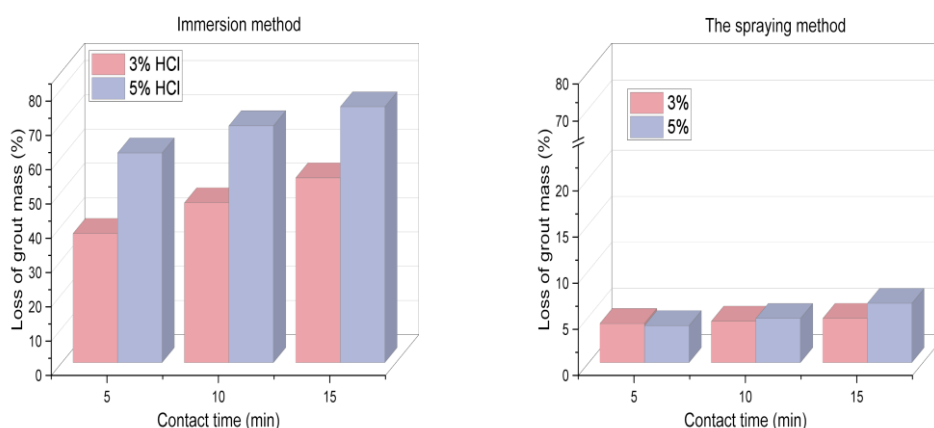


Figure 5. Comparative representation of grout mass loss between the immersion method and the spraying method

Fig. 5 shows significant dissolution of the grout using the immersion method, which directly depends on the concentration of HCl and the contact time between the acid and the grout. The spraying method shows considerably lower level of dissolution, which is less influenced by the concentration of HCl and more by the contact time. Using 3 w/w% HCl with the spraying method results in a grout mass loss of 4.2% to 4.8%, while using 5 w/w% HCl yields a grout mass loss of 4.0% at a contact time of 5 min and 6.4% at a contact time of 15 min.

The reason for the different behaviour of grout mass loss when applying different methods can be found in the micro-porosity of cement, an essential component of inorganic adhesives [17]. The pore structure of hardened cement paste is multi-component. According to [18], there are four types of pores present in hardened cement paste: gel pores (<10 nm), small capillary pores (10–100 nm), large capillary pores (100–1000 nm), and air pores (> several μm). The formation of dominant degradation pathways for the grout depends on the relative size of the micropores. Larger pores can accommodate a greater amount of HCl, which is facilitated by the immersion method, resulting in a significantly greater mass loss. However, in the spraying method, the amount of HCl that enters the pores is reduced, leading to lower mass loss.

The results of the conducted research need to be applied in the spa complex to confidently establish that the use of 3 w/w% HCl or 5 w/w% HCl achieves satisfactory cleaning results for tiles from calcium carbonate deposits. This approach not only prevents significant damage to the tiles due to the use of a less aggressive cleaning agent but also leads to cost

savings, as smaller quantities of chemicals are consumed per cleaning cycle.

CONCLUSION

The chemical analysis of the water from Banja Kulaši showed that it is thermal (28 °C), highly alkaline (pH = 11.85) water, with low mineral content, which can be classified as soft water based on its total water hardness (101.00 mg $\text{CaCO}_3/\text{dm}^3$). The chemical composition of the scale that forms as a result of the cooling of the thermal water on the edge tiles of the pool revealed that the dominant component of the scale is CaO (52.82 w/w%), which classifies this scale as carbonate scale. Based on the chemical analysis of the grout used for tiling, it was determined that, in addition to CaO (39.59 w/w%), the grout also contains silicates (16.11 w/w%), which is why the dissolution with HCl solution acts less aggressively on the grout compared to the dissolution of the scale.

The tests of the effectiveness of scale dissolution in the applied concentrations of citric acid did not yield satisfactory results.

The study of scale solubility using either the immersion or spraying method results in nearly identical effectiveness (>90%), with little influence from the concentration of the applied acid (3%, 5%, 10%). However, the solubility testing of the grout shows a dependence on the applied concentration of HCl, as well as a partial dependence on the applied contact time.

Use of 3 w/w% HCl achieves a scale dissolution efficiency of 91.67%, while the solubility of the grout in this case is minimal (4.01%). Based on the presented results for scale dissolution efficiency with different concentrations of hydrochloric acid, it can be

concluded that 3 w/w% HCl or 5 w/w% HCl, when applied by spraying for a contact time of 5 min, may represent an optimal solution that should be validated in the spa complex. This approach utilises a less aggressive acid solution compared to the previously used 10 w/w% acid, leading to savings as less acid is consumed per cleaning cycle.

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