CRYSTALLIZATION FOULING IN DOMESTIC APPLIANCES AND SYSTEMS

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Abstract

Formation of mineral scale, particularly calcium carbonate, has long been a concern in domestic appliances such as boilers, steamers and washing machines. In this work, the formation of inorganic deposits was studied on solid surfaces and in the bulk solution as a result of evaporation of potable water. Scale precipitation was evaluated on metallic samples for various bulk temperatures, surface roughness and surface material as a function of time. The morphology of scale crystals from both surface and solution was evaluated. The microstructure and morphology of deposits on the surface were analysed using Scanning Electron Microscopy and X-ray diffraction. The present work has demonstrated that deposition rates increase as the evaporation process proceeds as expected. The nature of the material and the surface roughness both affect surface deposition kinetics. The complex relationship between the saturation ratio (as the bulk solution shows precipitation) and the kinetics of bulk and surface precipitation/deposition is demonstrated. The prevailing crystalline form of calcium carbonate is aragonite. However, the polymorphic composition of surface deposition and the bulk precipitate is influenced by temperature.

Introduction

The scale is often defined as the unwanted deposition of minerals on a heat transfer surface. It is an extremely common and complex problem in industrial and domestic systems where potable water is a working fluid [1, 2]. The precipitated layer of inorganic salt generally comprises calcium carbonate, calcium sulphate, magnesium hydroxide, etc., generated by a chemical reaction of cations and anions in a solution. An increase of the solution temperature promotes the reaction kinetics, nucleation and growth rate of a sparingly soluble inorganic scale [3, 4]. The resistance to heat transfer caused by scale fouling costs money by diminishing the efficiency of heat transfer. It also boosts the pressure drop in systems by reducing the flow area and increases maintenance cost and replacement works [5-8].

Fouling of heat transfer surfaces in domestic appliances results in environmental and economic impacts. The power consumption of electric boilers, steam irons, washing machines, dishwashers and coffee makers is significantly increasing due to the retarded thermal conductivity of fouling layer [9]. It is estimated that the heat transfer efficiency of gas water-boilers will be reduced by 30% over a period of 15 years if no effective mitigation strategy is applied [10]. Dobersek and Goricanec [11] investigated the influence of the precipitated water scale on the consumption of electrical energy for domestic appliances. They found that scale layers of 2 mm thickness reduced the heat flow by 10% to 40%, depending on the composition of scale. For the same scale thickness, the power consumption of an electric boiler is two times higher than that for a washing machine.

Temperature is one of the decisive crystallization parameters which can influence both saturation degree, reaction kinetics, and scale morphology. In the work of Yang, et al. [12], the effect of surface temperature on scale formation on the copper-modified surface has been examined. The temperature increase from 70.8 ºC to 82.8 ºC resulted in fouling resistance increase by more than

7 times. The presence of mineral ion traces in water may pose an impact on the growth process of scale crystals. Chen, et al. [13] reported that the kinetics of $CaCO_3$ bulk precipitation for different concentrations of magnesium ions using rotating disk crystalliser (RDC). The results showed that Mg^{2+} has an inhibitory effect on the bulk precipitation as well as $CaCO_3$ polymorphic phases.

The surface roughness was shown to exert a remarkable impact on the tenacity of the calcium carbonate deposits. Keysar, et al. [14] pointed out the shear stress for detaching the calcite crystals binding to the rough surface (R_a= 18-24 µm) was estimated to be as much as 30 times greater than that for smoother surface (R_a=0.1-0.15 µm). The results also revealed that the scale formed on the smooth surface was three times higher in porosity than that on the rougher one.

The pipe material is one of the quality criteria in a domestic appliance that represents heat transfer efficiency. The commonly used metals for this purpose are stainless steel, copper and aluminium. Substrate parameters such as surface energy and thermal conductivity could influence the scaling tendency, adhesion and morphology of scale [15, 16]. Teng, et al. [17] studied the surface deposition rate on copper, aluminium, stainless steel, brass, and carbon steel. The highest amount of scale was deposited on copper while the lowest amount was deposited on stainless steel.

Investigations into crystallization fouling have largely focused on the surface deposition in the industrial systems using solutions supersaturated with calcium and carbonate ions. The supersaturation of similar solutions at constant temperature decreases with time due to consumption of ions in the crystallization reaction. The water heating in household devices mostly includes water evaporation. Aspects of the mechanism of the scaling process from tap water in domestic appliances under evaporation conditions are still not fully understood. Evaporation of test solution, besides Ca^{2+} and CO_3^{2-} consumption, affects solution supersaturation. In the present study, the scale formation from potable water has been investigated to improve the understanding of fouling mechanisms in domestic systems. The effects of solution temperature, surface roughness and surface material on the scale amount and morphology have been examined.

Materials and methods

Experimental setup

Experiments are performed in a unique setup mimicking a batch evaporation system. The schematic drawing of the setup is presented in figure 1. It consists of a borosilicate glass beaker, hot plate, thermocouple, sample holder, and identical metallic samples. The sample holder is constructed of stainless steel 316L (SS31603) for minimizing the potential of corrosion products formation. The temperature probe (ThermoScientific, UK) was connected to a laboratory hot plate to control the temperature in a 1000 ml borosilicate glass vessel. Eight cylindrical samples of each copper C12200, aluminium 1050A and stainless steel SS31603 have been made with similar height and diameter of 10 ± 0.05 mm. The samples are fabricated with a hollowed bottom with a depth of 8 ± 0.05 mm to be established on the sample's holder vertical fines.

Using artificial hardened solutions for studying the scaling process may not provide a full understanding of fouling mechanisms in domestic systems. The presence of different ionic species in potable water may affect the fouling rate and scale morphology. The commercially available *Evian* bottled water has consequently been used as a scale-forming solution. As *Evian* being very hard water with a hardness of 307 ppm of $CaCO_{3,}$ it has been chosen for the test rather than tap water to accelerate scale formation. The diversity of ionic species in drinking or tap water may pose some influences on the fouling phenomenon. The composition of *Evian* water is listed in Table 1.

Stainless steel samples with the mirror-like surface have been used in the experiments to evaluate the temperature effect. The tested temperatures are 85, 95, and 105 ºC. The power consumed by the hot plane is ranging from 1748 to 2139 W for the tested temperatures. The rate of evaporation of water increases with water temperature due to an increase in the kinetic energy of molecules (Fig. 2). In the investigations of substrate effect, the three metallic surfaces; copper, aluminium, and stainless steel have been tested at constant roughness and bulk temperature of 95 ºC. Stainless steel samples subsequently used in the roughness experiments at a fixed bulk temperature of 95 ºC. Roughness average (R_a) is the roughness parameter adopted in the current work. The tested surface roughness values are 10, 106, and 182 nm. To check the reproducibility of all experiments, the runs are repeated at least twice.

Surface and solution characterization

The top surface of the sample was grinded with silicon carbide paper (1200 grit) and then polished with diamond suspension (0.5 μm) by a mechanical polishing method to obtain surface roughness (Ra) of 8-24.6 nm. Surface roughness parameters were evaluated prior to the experiments using the 3D optical profiler NPFlex (Bruker, USA). For every 100 ml of the evaporated water, a sample and 1 ml of solution were taken from the test vessel. The solution volume was mixed with 9 ml of a quenching (KCl/polyvinyl sulfonate) solution to prevent further precipitation. Quenching solution was prepared by adding 1 mg of polyvinyl sulfonate (PVS) scale inhibitor and 5.71 gm of potassium chloride (KCl) in 1000 ml of distilled water.

The analysis of Ca^{2+} concentration of the resultant solution was carried out using the Atomic Absorption Spectrophotometry (AAS). The measured concentration of Ca^{2+} at different evaporated volumes was introduced to calculations of saturation ratio. The samples were dried at 37 ºC for 24 hours, followed by scale mass measurement by a high accuracy lab balance. The deposit morphology was examined using a Philips X'Pert X-ray diffractometer (XRD) (X'Pert MPD, Cu anode x-ray source, Netherlands) and Scanning Electron Microscope (SEM) (Carl Zeiss EVO MA15). Elemental analysis of the deposits was carried out using an Energy-dispersive X-ray (EDX) (Oxford Instruments AZtecEnergy) with an accelerating voltage of 15 kV.

Results and discussion

Effect of bulk temperature

Figure 3 shows the amount of scale precipitated on the stainless steel surface increases with the evaporated water volume and the bulk temperature. Temperature increase from 30 to 90 ºC reduces the solubility of $CaCO_3$ from 220 to 40 mg/L and hence increases the supersaturation and scaling tendency at a constant concentration of foulant [18]. The consumption of calcium ions in water by crystallization reaction increases with the volume of evaporated water at 95 ºC (Fig. 4). It can be seen from the exponential decay of calcium ions, the consumption rate of calcium in the solution is faster than the water evaporation rate, as such saturation ratio decreases with time, as presented in figure 3.

The SEM observations in figure 5 show that the needle-like aragonite crystals are the dominant form of $CaCO_3$ at all tested temperatures with various amounts of mucus-like particles. The elemental analysis by EDX shows that magnesium content in the flake-like particulate is slightly higher than that in aragonite particles (Spectrums 6 and 7 in Fig. 6). However, the magnesium may have an inhibitory effect on the morphological transformation to crystalline particles [19-21].

The XRD analysis of scale deposited on the surface illustrates that the aragonite is the prevailing polymorph $CaCO_3$ for all temperatures (Fig. 7). The solution temperature promotes the morphological transformation on the surface from vaterite and calcite to aragonite. In the XRD for precipitates in the bulk, a few changes have been spotted; namely, the transformation of aragonite to calcite, and the presence of calcium sulfate as anhydrite at temperatures of 95 and 105 ºC (Fig. 8).

Effect of surface material

The visual observations show that the aluminium sample was coated by a black layer. According to the SEM-EDX analysis, this porous black crust comprises Al and O, and this strongly suggests the formation of aluminium oxide (Al_2O_3) layer (Fig. 9). No significant variation in the amount of the precipitated scale among the tested surfaces has been observed (Fig. 10). The mass gain on aluminium is a mixture of oxide layer and mineral deposits. As crystal forms in the bulk solution by the homogeneous nucleation, the surface may not affect the nucleation and growth process. The surface nature nevertheless can influence the adhesion with the precipitated crystal [22].

The common denominator among the morphology of the deposits on the different surfaces is the presence of flake-like particles which occupy a large area of the copper surface compared to the other metals. The copper ions yielded as a corrosion product may inhibit the transformation from amorphous to crystalline form [23]. Al^{3+} also may behave similarly on the aluminium substrate but with lower inhibitory efficiency as a consequence of consumption of ions in the oxidation reaction. It seems furthermore that the population of scale crystals on aluminium is relatively low and the oxide layer may provide a weaker adhesive strength. Regarding the scale structure on stainless steel, the secondary growth of the flake-like particulates might heterogeneously commences after the aragonite binding on the surface.

The XRD analysis shows differentiated findings in terms of abundance of the crystalline phase's, crystal shape, and the analysis intensity (Fig. 12). The $CaCO_3$ vaterite on copper is higher than that on the other materials. The absence of ionic species such as Al^{3+} and Cu^{+} may allow the transformation to aragonite form. Crystals of γ-alumina (γ-Al_2O_3) have been detected on aluminium, which confirms that the dark porous crust underneath the deposits is built of aluminium oxide.

Effect of surface roughness

Figure 13 shows that the precipitated mass on all surfaces is very similar, particularly in the earlier stages of the evaporation. It seems that it is hard to remove the scale particles from the rougher surface by the turbulent bubble circulation at the later stages of evaporation, where the water level is low. As the crystals are formed by homogenous crystallization, the influence of roughness concentrates on the aspects of particle-surface adhesion. The SEM observations in figure 14 show that the amount of deposit flakes increases with the roughness. It also illustrates that the flowers of aragonite formed underneath the scaly coat. However, the presence of these flakes can affect the accuracy of the XRD analysis. Many differences can be observed in the XRD measurements related to peaks intensity and position, and the detected polymorphs (Fig. 15). The significant morphological transformation paths include calcite on the smooth surface to aragonite on the moderately rough surface (R_a= 106 nm) which in turn transforms sequentially to vaterite particle on the rougher surface (R_a= 182 nm).

Conclusions

Evaporation of potable water in domestic appliances has generally been associated with precipitation of deposits in the bulk solution that binds onto appliance hot surfaces. The present work investigates the crystallization fouling with water evaporation process at different solution temperature, surface material, and surface roughness. The deposited mass predominantly increases with the volume of evaporated water, while Ca^{2+} solution content simultaneously decreases according to an exponential decay relationship. The solution temperature promotes the bulk precipitation and the polymorphic transformation from both calcite and vaterite to aragonite. It is found that flake-like particles were formed above the crystalline particles.

The amount of flake-like deposits increases with temperature and it consists of about 4% by weight of Mg. At the high temperatures, Mg^{2+} in water may interact with growing crystals and inhibits a further growth to aragonite. Similarly, the scale structure on aluminium and copper surfaces might be affected by the presence of Cu^{2+} and Al^{3+} which released as corrosion products.

The effects of the surface substrate and roughness were found to be secondary on the precipitated mass. As the crystal forms in the bulk solution by the homogeneous nucleation, a surface may not control the nucleation and growth process. The surface nature nevertheless can affect the adhesion force with the precipitated crystal. The morphology analysis showed that calcite on the smooth surface turns into aragonite on the surface with moderate roughness and aragonite to vaterite on the rough surface. The results obtained in the present work is dependent on water chemistry.

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Nomenclature

Abbreviations

Al_2O_3	Aluminium oxide
A	Aragonite
AAS	Atomic Absorption Spectrophotometry
C	Calcite
$CaCO_3$	Calcium carbonate
EDX	Energy Dispersive X-Ray
KCl	Potassium chloride
PVS	Polyvinyl sulfonate
Ra	Average Roughness
RDC	Rotating disk crystalliser
S	Calcium Sulfate Anhydrite
SEM	Scanning Electron Microscope
V	Vaterite
XRD	X-Ray Diffraction

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Table 1. Evian drinking water composition.

Ion	**ppm**
Ca^{2+}	80
Mg^{2+}	26
Na^{+}	6.5
K^{+}	1
Si^{4+}	15
HCO^{3-}	360
SO_4^{2-}	14
Cl^{-}	10
NO^{3-}	3.8
Dry residue at 180ºC	345

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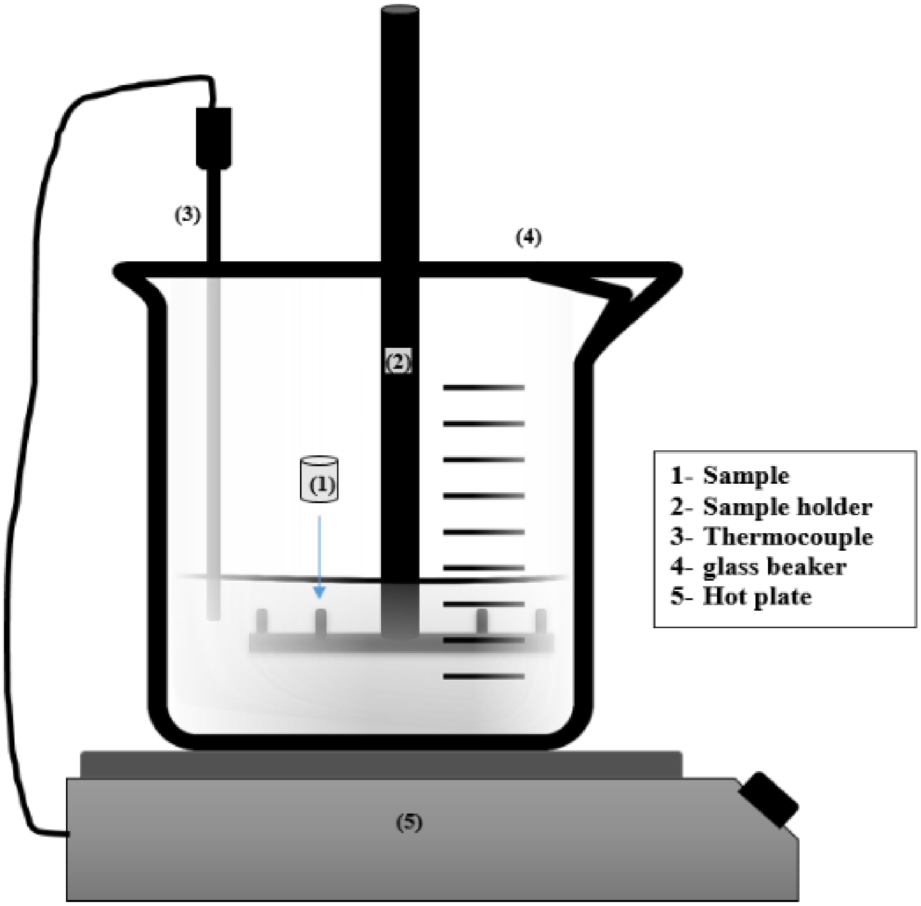


Figure 1. Schematic of the test setup.

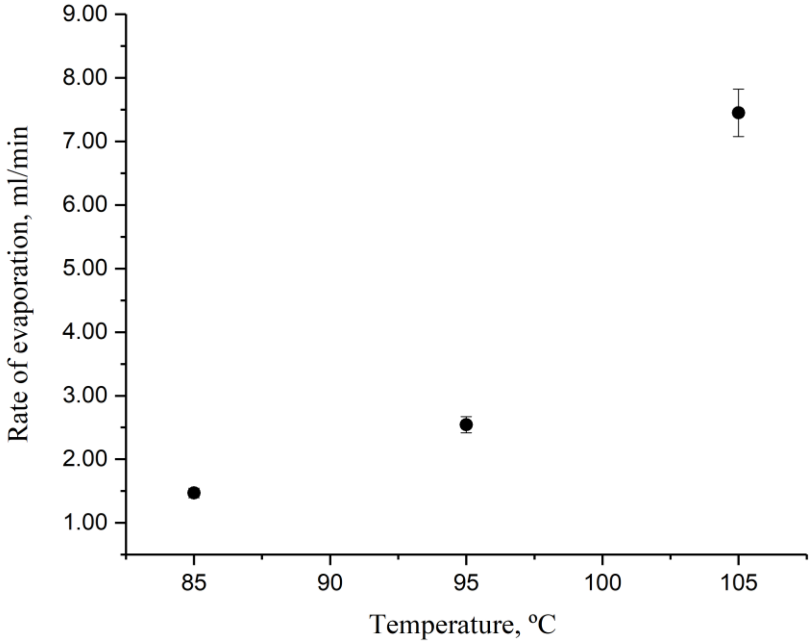


Figure 2. The evaporation rate of solution at different temperatures.

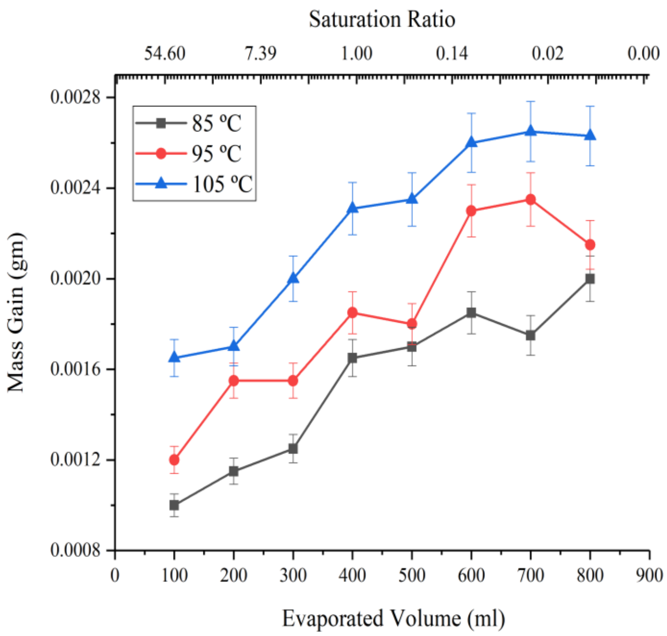


Figure 3. Effect of temperature on the scale formation on stainless steel.

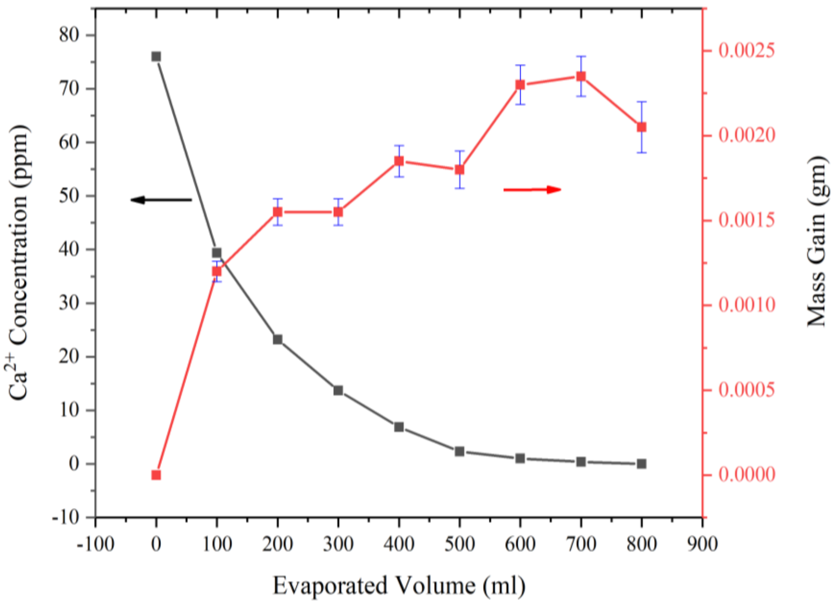


Figure 4. Effect of water evaporation on Ca^{2+} concentration at 95 ºC.

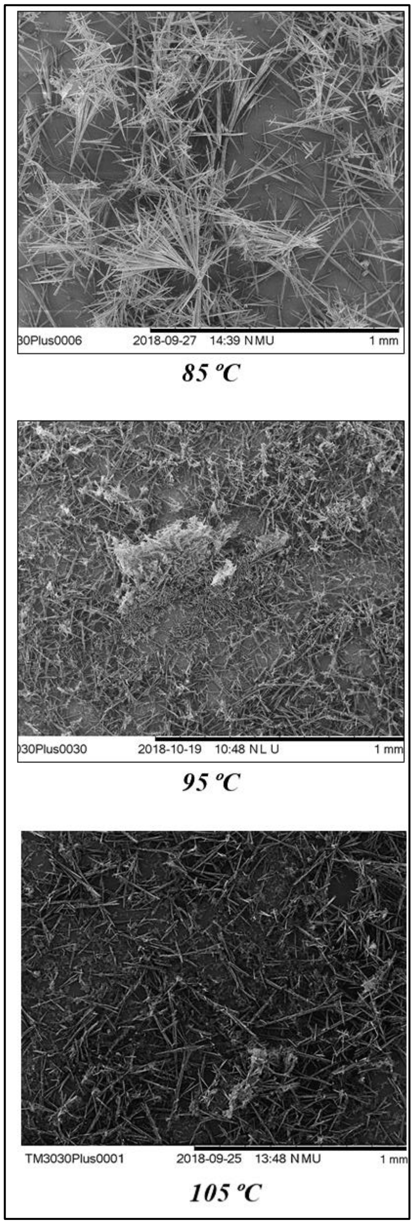
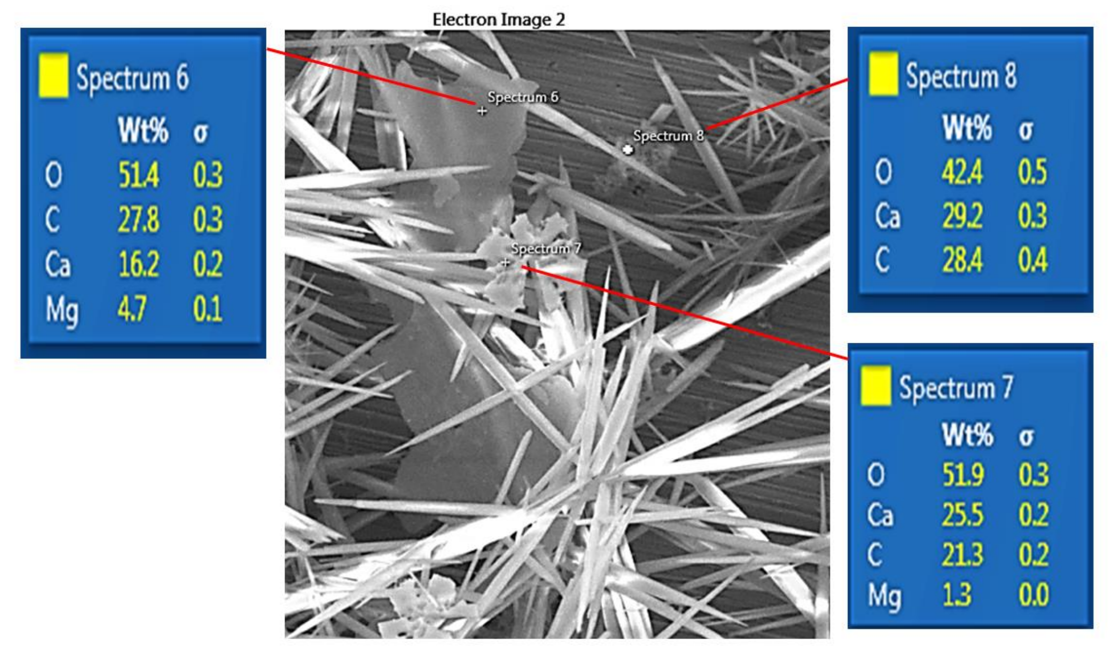


Figure 4. SEM observations of scale crystals for a different solution temperature.



Spectrum 6

	Wt%	σ
O	51.4	0.3
C	27.8	0.3
Ca	16.2	0.2
Mg	4.7	0.1

Spectrum 8

	Wt%	σ
O	42.4	0.5
Ca	29.2	0.3
C	28.4	0.4

Spectrum 7

	Wt%	σ
O	51.9	0.3
Ca	25.5	0.2
C	21.3	0.2
Mg	1.3	0.0

Figure 6. EDX elemental analysis for different forms of calcium carbonate deposits.

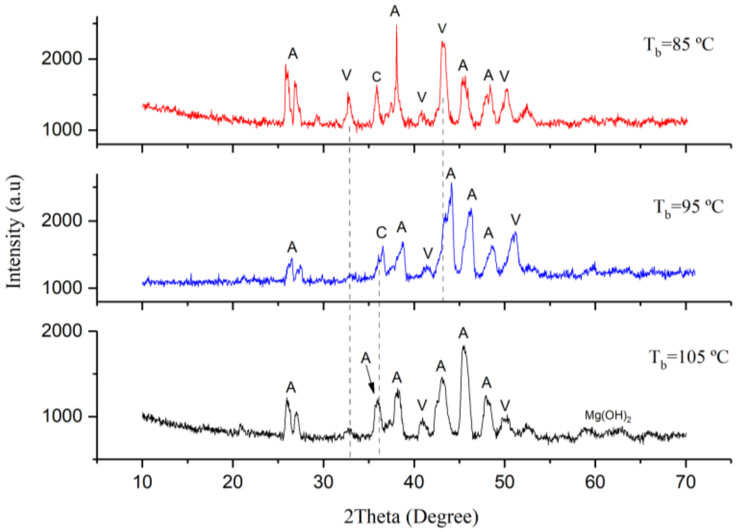


Figure 7. XRD analysis of the scale crystalline species on the surface (A: Aragonite, C: Calcite, and V: Vaterite).

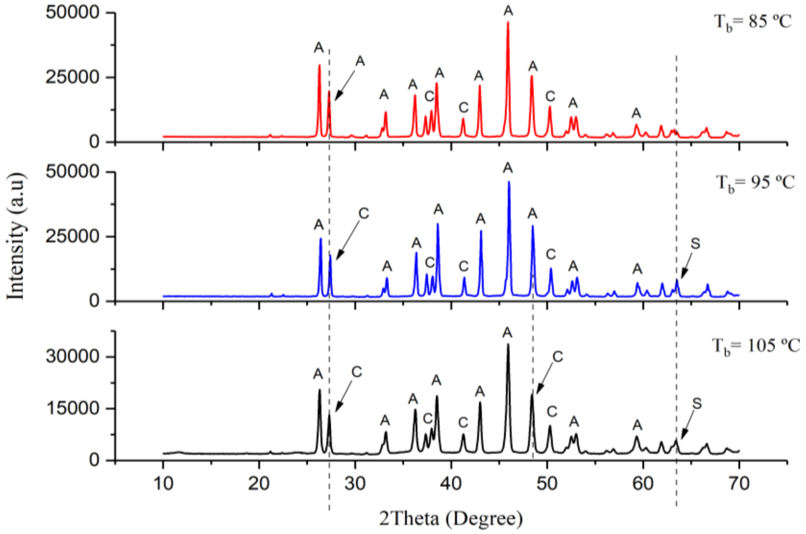
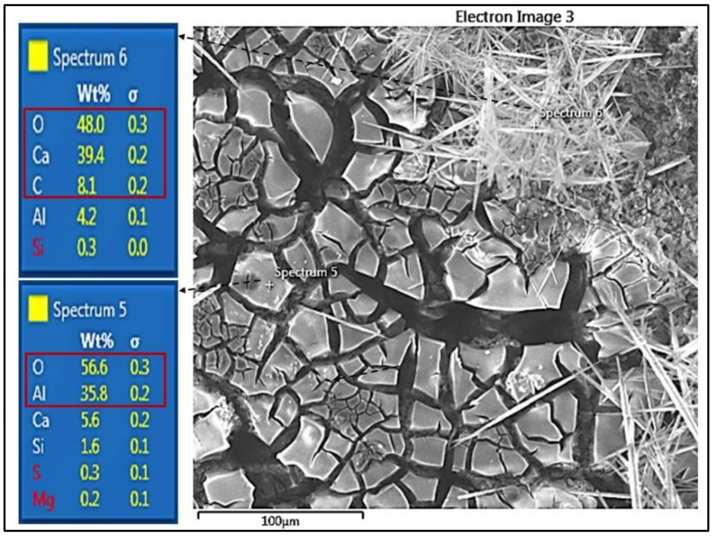


Figure 8. XRD analysis of the bulk precipitation (A: Aragonite, C: Calcite, and S: calcium sulfate as anhydrite).



Spectrum 6

	Wt%	σ
O	48.0	0.3
Ca	39.4	0.2
C	8.1	0.2
Al	4.2	0.1
Si	0.3	0.0

Spectrum 5

	Wt%	σ
O	56.6	0.3
Al	35.8	0.2
Ca	5.6	0.2
Si	1.6	0.1
S	0.3	0.1
Mg	0.2	0.1

Figure 9. The EDX analysis for oxide layer and scale crystals on the aluminium surface.

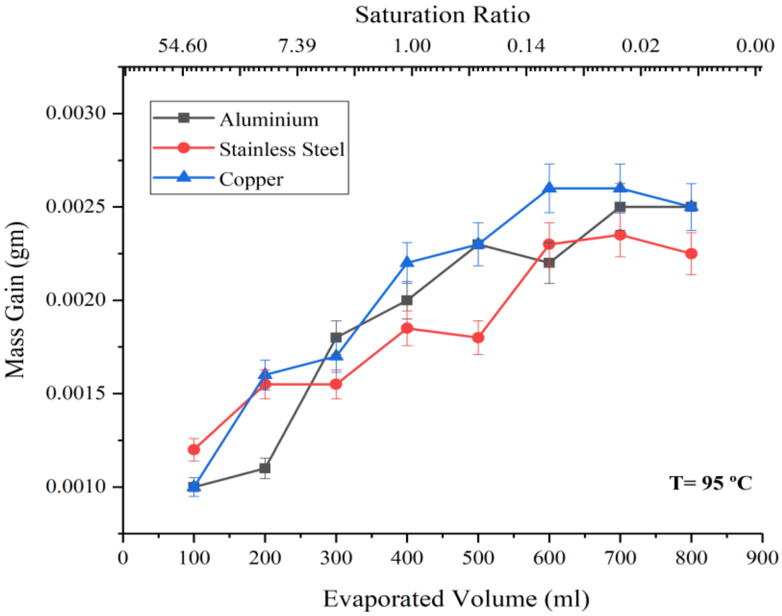


Figure 10. Effect of surface material on scale deposition.

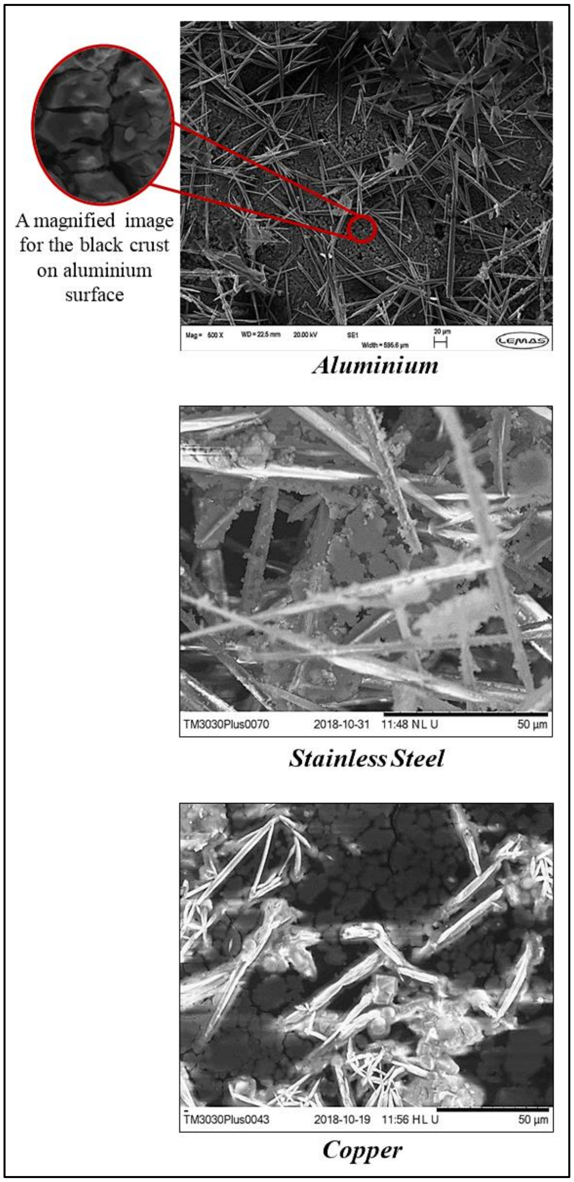


Figure 11. SEM images of scale particles on different surfaces.

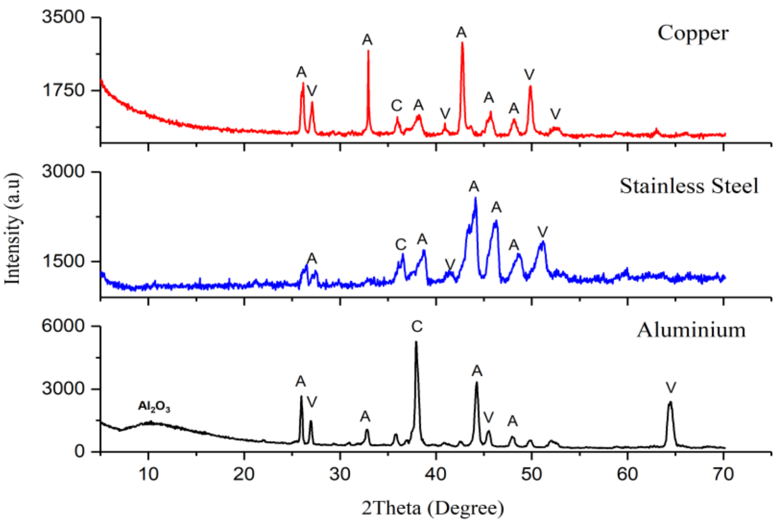


Figure 12. XRD analysis of scale crystalline species on different surfaces.

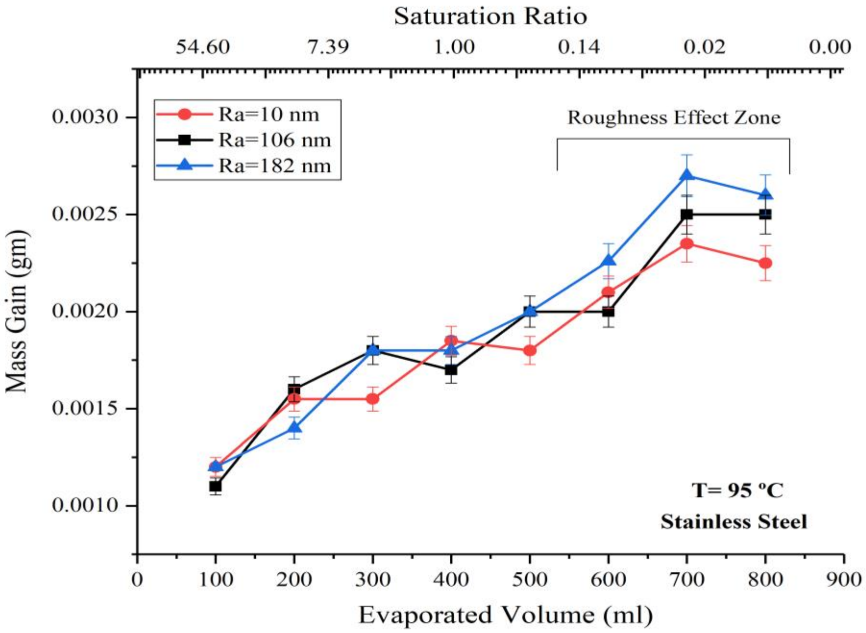
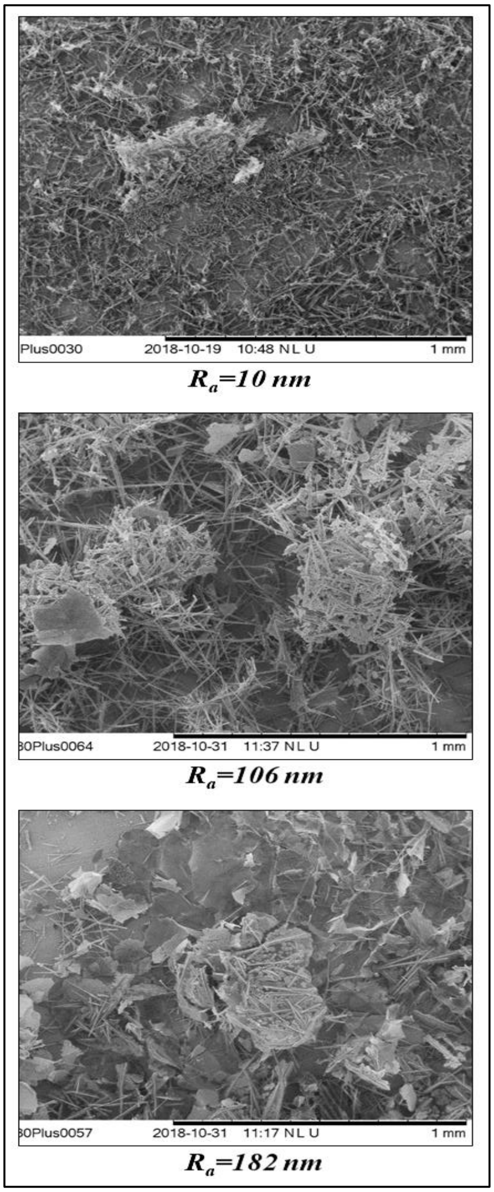


Figure 13. Effect of surface roughness on scale deposition.



R_a=10 nm

R_a=106 nm

R_a=182 nm

Figure 14. SEM images of scale deposits on surfaces with different roughness.

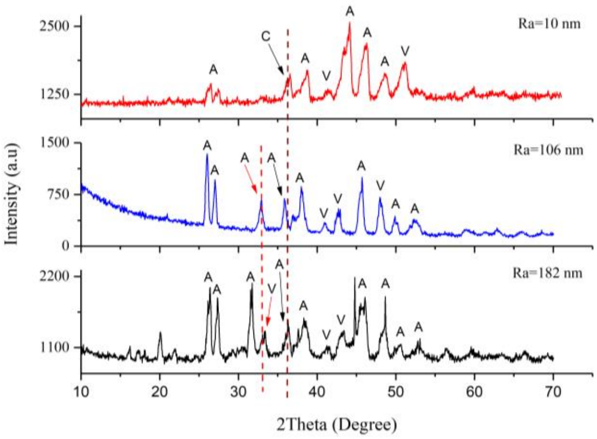


Figure 15. XRD analysis of the scale crystalline species on different roughness surfaces

Biographical Information

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