

Effects of Operation Conditions on Incrustation Phenomena

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INTRODUCTION

Scale formation on the heat transfer surfaces in horizontal tube evaporators leads to an additional heat transfer resistant which diminishes the process efficiency. For this reason scale prevention is of utmost importance. In horizontal tube evaporators, which are increasingly employed in multiple-effect distillation (MED) plants for seawater desalination, the scale is formed on the outside of the tubes. Therefore, effective scale prevention and cleaning are difficult to achieve.

Scale formation in MED seawater distillers is mainly caused by the crystallization of the inversely soluble salts calcium carbonate, magnesium hydroxide and calcium sulfate. To date a chemical treatment is the most promising prevention method. To create an appropriate tool to prevent incrustation the scale composition and the crystal structure in relation to operation conditions have to be known.

The main problems of researching this topic and to create authentic scales are to operate a test rig under real conditions (e.g. temperature, pressure) and the extremely long experiment time.

AIM

The aim of this work is to find optimal operation conditions where a satisfactory scale layer forms in an adequate time so that analyzing methods can be applied for an accurate characterization of the layers with respect to the composition and the crystal structures.

Structural and chemical information of the scales formed from falling films on horizontal tubes under conditions very similar to real process conditions in MED distillers are to be obtained in

order to get a better understanding of the crystallization phenomena and to improve scale control.

EXPERIMENTAL SETUP

Two falling film test rigs, one small size technical plant (laboratory style) was built and a second plant of semi-industrial size (pilot plant scale) was constructed. In both test rigs the test solution is distributed over a row of horizontal tubes and trickles down by gravity forming a film flow over the tubes. The lab scale plant is perfectly fit for fast and easy variations of process parameters and data collection while the scaling process is running.

In the laboratory test rig the product cycle is operated under ambient pressure. The test solution is only heated without evaporation. The adaption of the multi-effect distillation (MED) process to a small technical scale enables the variation of different process parameters like trickling rate and temperature profiles.

On the contrary the pilot plant – much more difficult and time consuming to operate - enables the simulation of the process conditions in any stage of an industrial MED plant such as pressure, temperature and evaporation conditions.

The tubes can be removed from the test rigs and thus the formed scale can be analysed. With respect to former research work and to applicable experimental time a duration of 50 hours was chosen. The surface material for all experiments is the stainless steel 304. This is an austenitic stainless steel which meets most of the requirements in the water industries, e.g. good combination of corrosion resistance, forming and fabrication properties [2]. The trickling rate is kept constant for all experiments.

TEST SOLUTION

Due to the practical background of the seawater desalination industry and the high salinity, artificial seawater was chosen as an adequate test fluid. The used solution has the composition of standard seawater with a salinity of 35 g/kg, according to the formulation suggested by Riley and Chester [1], shown in Table 1. The preparation of seawater with higher salinities simulating the seawater in the Arabian Gulf and/or concentrated brines in the distiller was based on the formulation shown in Table 1.

Salt	g/kg
NaCl	23.926
Na ₂ SO ₄	4.008
KCl	0.677
NaHCO ₃	0.196
KBr	0.098
H ₃ BO ₃	0.026
NaF	0.003
MgCl ₂ *6H ₂ O	10.83
CaCl ₂ *2H ₂ O	2.75
SrCl ₂ *6H ₂ O	0.013

Table 1: Preparation of artificial seawater with a salinity of 35 g/kg [1]

STRUCTURAL CHARACTERIZATION OF SCALES

The structural characterization of scales generated in the plants was performed at the laboratories of BASF. First of all Scanning Electron Microscopy (SEM) in combination with Energy Dispersive X-Ray Spectroscopy (EDXS) and Wide Angle X-Ray Diffraction (XRD) were applied in order to obtain structural and chemical information about the scale on length scales from nanometers to millimeters (crystal size and orientation, composition, crystal structure, crystal perfection, etc.).

An advantage of SEM is the ease of preparing the samples and specimens of several cm in size can be examined at magnifications of 10 x for a general view, up to 100k x for a detailed view. Aside of secondary electrons X-rays released from the surface elucidate the local elemental composition. The energy of the radiation analyzed by means of a Si(Li) – Diode allows identification of elements and from the signal strength local elemental concentration ratios within the field of view can be calculated. On base of this type of information the nature of imaged structures can be allocated.

The determination of crystalline phases can effectively be accomplished by diffraction methods. The applicability of the wide-spread conventional XRD however might lack the sensitivity for detecting the rather small amount of crystalline material in the early stage of a scale build-up. Benefits were taken from the most modern generation of XRD-instrumentation exploiting an ultimate parallel beam concept. Instead of sacrificing beam intensity for suppression of the fluorescence background radiation and for improved monochromacy this technique utilizes a parallel X-ray beam generated by a parabolically bent so-called Göbel Mirror which is directed

to the surface covered with scale. The angular intensity distribution of the Bragg reflected primary beam offers an enhanced peak-to-background ratio and due to that a sensitivity which allows us studying the growing scale layer from the very beginning on [4].

RESULTS

In the presented results the parameters salinity, heating temperature and evaporation temperature are varied.

The first experiments show the effect of the variation of salinity. In Figure 1 SEM pictures are showing the beginning of the incrustation process. The tube surface is partly covered with crystals after an experiment duration of 50 h. Mainly rod-shaped aragonite crystals of uniform size about 10 µm were found on the tube surface. These experiments have been carried out at different salinities. The optical analyzation shows no significant difference by variation of the salinity, as shown in Figure 1a) and b).

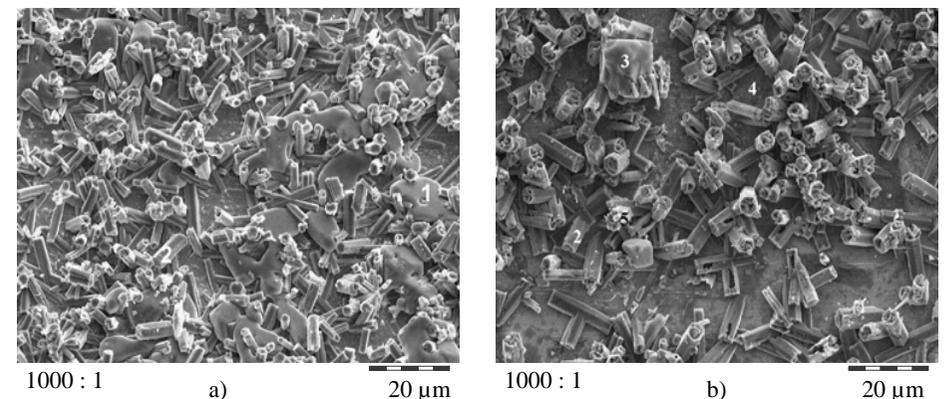


Figure 1: SEM showing crystals formed at same heating and evaporation temperature with test solutions of a salinity difference of 10 g/kg.

An analyzation of the samples by XRD also does not show any difference in structure and chemical composition.

Further experiments have been carried out for other temperature levels with a constant salinity. The results are presented in Figure 2. In Figure 2b a higher coverage of the tube surface is reached by expanding the experiment time to 100 h but there is also no significant change in crystal structure and chemical information.

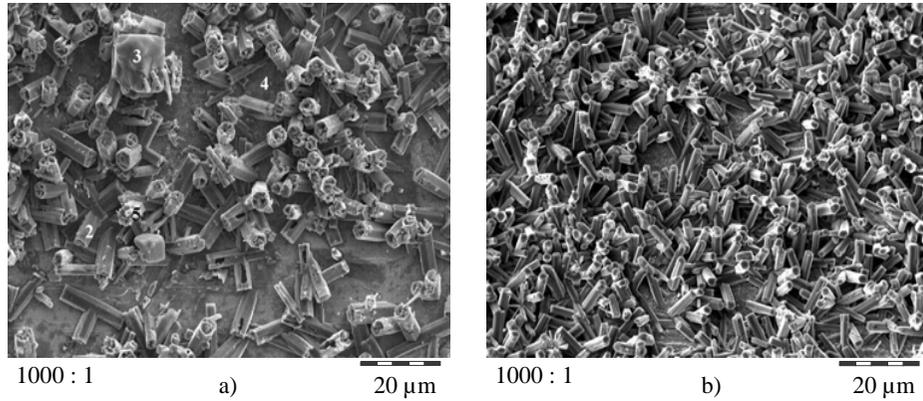


Figure 2: SEM showing crystals formed at different heating and evaporation temperature levels with the same test solution.

The results shown in Figure 1 and 2 were gained by constant heat transfer data like heat flux and heat transfer coefficient. To increase these data a higher temperature difference between heating and evaporation temperature was chosen. By this action not only the coverage of the heat transfer surface with crystals was increased but also the crystal structure was significantly changed. At lower ΔT a crystal growth occurred but by increasing ΔT only an agglomeration of different salts take place and forms totally different structures.

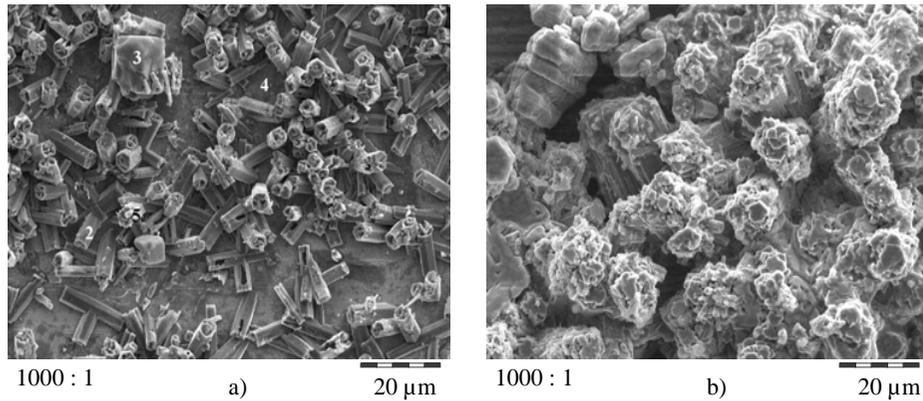


Figure 3: SEM showing crystals formed at different ΔT between heating and evaporation with the same test solution.

A closer look at higher magnifications shows four types of crystals of different chemical compositions which occur on the outer surface of the heat transfer area under different operation conditions. These four types are: big cubic crystal (Figure 4a), small rod-shaped crystals (Figure 4b), agglomerates of different components (Figure 4c) and very small particles of a size less than $1\mu\text{m}$.

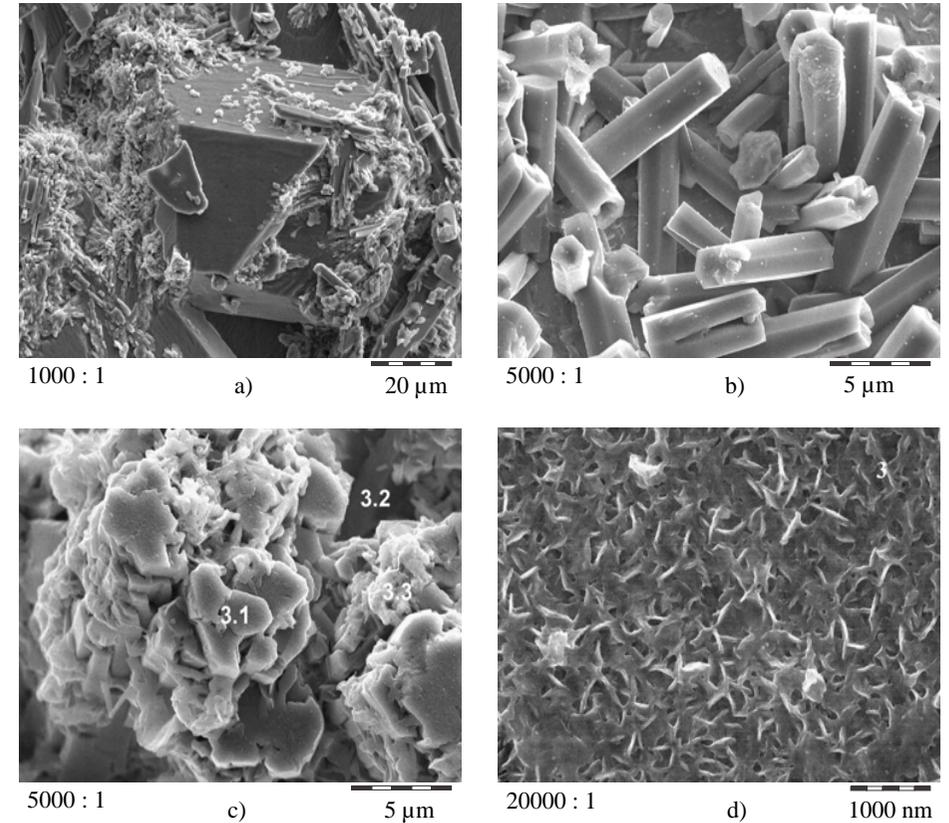


Figure 4: SEM showing the variety of occurring crystals during the evaporation process under varying operation conditions.

In the laboratory plant the temperature difference and the heat flux are always high compared to the pilot plant. But the overall heat transfer coefficient ranges in the same level as in the pilot plant. The sample tubes from the laboratory test rig show only a very thin incrustation and less

coverage of the surface. The incrustation from the laboratory test rig shows similar structures and chemical compositions like incrustations from the pilot plant. This is shown in Figure 5 at varying magnifications.

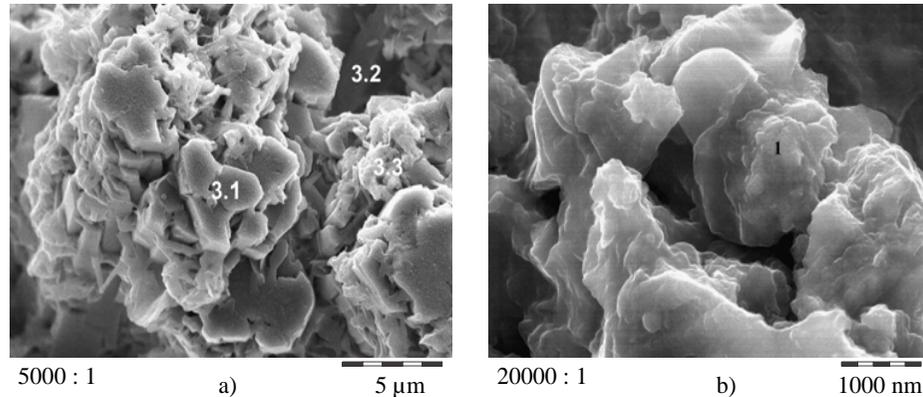


Figure 5: SEM showing crystals formed at high ΔT between heating temperature and evaporation temperature. a) from the pilot plant and b) from the laboratory plant

CONCLUSION

The experiments with seawater as a test solution and the selected experimental time of 50 hours show satisfactory results of incrustation. The expected inversely soluble salts crystallize on the surface of the heat transfer area.

The presented experimental results, which are analyzed by XRD, show no significant variation of the chemical composition. In addition to that a variation of the salinity just as a variation of the temperature level of heating and evaporation temperature without significantly increasing the heat flux cause no structural change of the crystals. Only a higher coverage can be achieved by extending the experiment duration to 100 hours. However, there is a well noticeable difference of the crystal structure which strongly depends on operation conditions. Increasing the temperature difference between heating and evaporation temperature, this means also increasing the heat flux, the crystal structure totally changes. The formation behaviour of the crystals changes from crystal growth to randomly agglomeration of the precipitated salts. The same effect can be observed in the laboratory test rig which is always operating with a high heat

flux. The thickness of the layer and the coverage of the tube surfaces from the laboratory test rig are less and therefore are more difficult to analyze. This shows that the pilot plant is needed to produce reliable and reproducible scale layers. Furthermore a wide variety of crystal structures in particular at lower temperature differences, which are closer to real operation conditions can only be gained from the pilot plant.

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