Selection of Materials for the Detection of Biofouling in Cooling Water Systems

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Abstract

In cooling water systems there are several issues, such as corrosion and fouling, which decrease the process efficiency and increase the costs associated. The main objective of this work consisted in the selection of materials capable of favouring the formation of biofilm for its early detection in real-time by the Diveil Surface Sensor (DSS). Another goal was the development of plane coupons for the early detection of biofouling, in alternative to expensive commercial coupons.

In a lab set-up, DSS monitored the biofilm adhesion on two sensor tubes, one of stainless steel (SS) and another of polyvinyl chloride (PVC), inside which flowed water collected from a cooling system of a food industry. The biofilm adhesion was also quantified inside cylindrical coupons of the same materials and diameters to simulate DSS sensor tubes. In the assay performed, the sensor tube and the cylindrical coupon in stainless steel led to the more satisfactory results, i.e., the greatest vibration amplitude and mass of adhered biofilm, respectively.

As for the plane coupons, distinct materials were tested: copper, SS, PVC, high density polyethylene (HDPE) and neoprene. In the assay performed, the neoprene revealed the highest mass of adhered biofilm, although HDPE and PVC showed effective responses and are corrosion-free.

The characterization of all materials tested was performed through scanning electron microscopy (SEM) and goniometry, and the conclusion drawn was that the surface shear stress had a strong influence in the biofilms adhesion followed by the surface effective roughness and hydrophobicity.

Keywords: Biofouling, Coupons, Copper, HDPE, PVC, Stainless steel.

1. Introduction

In industrial applications, water is commonly used to refrigerate products and / or processes due to its high heat transfer capacity in order to maintain the constancy of the operating conditions relevant to the process efficiency (e.g., temperature and pressure) [1].

Moreover, it is very well-known that water is an universal solvent. In industry, this feature is not always desirable because water may dissolve many substances, including gases, for instance oxygen and carbon dioxide that favour metals corrosion. As water also dissolves minerals, their concentrations may exceed their solubilities and lead to scaling. Furthermore, water bears nutrients providing ideal conditions for microbiological growth, i.e., biofouling [1].

The designation ‘fouling’ stands for the undesirable formation of inorganic and / or organic deposits on the materials surfaces. These deposits decrease the heat transfer across the surfaces, and increase the fluid pressure losses and the corrosion rates on the surfaces, each of these phenomena producing energy losses. There are several types of fouling [2]:

1) scaling due to minerals precipitation;
2) corrosion due to corrosion reactions;
3) organic fouling,
4) biofouling.

![Biofilm development stages](image-url)
Biofouling development is sketched in Fig. 1. In the first stage, a reversible adsorption of the cells occurs on the material surface by electrostatic attractions. The second stage includes the binding of the initial adsorbed cells by extracellular matter (produced by cellular metabolism) and medium nutrients, allowing the adhesion of new cells onto the surface. On the third stage, the initial cells reproduce, micro colonies are created, and the adsorption becomes irreversible. In the fourth stage, the biofilm maturation begins, and the biofilm density and complexity increase as the cells split. In this stage, the biofilm is fully hydrated and becomes slippery. In the fifth stage, the biofilm reaches its critical mass and the outer layers release cells that spread away to colonize new surfaces elsewhere [10].

In general, the microorganisms growth depends on the type of substrate for colonization, the physical conditions (temperature, pH, solar exposure, pressure, etc.) and the nourishment conditions (water, oxygen, CO₂, nitrogen, phosphorus, etc.). Usually, bacteria grow at about 20 – 40 °C but some species develop at 4.5 – 70 °C. The optimal pH of most microorganisms is around 7 [7][8]. The biofilm development is also influenced by several other factors, e.g., the materials properties, and the medium and bacteria characteristics [9].

The chemical properties that most influence microorganism adhesion are [10]:

1) the chemical composition, that affects the microorganism adhesion and proliferation, due to distinct hydrophobicity and electrical charges of the functional groups;
2) the surface roughness, as the greater the surface irregularities are, the higher the biofilm growth rate is, due to the higher surface area available for biofilms adhesion.

In general, the microorganisms prefer to adsorb on hydrophobic nonpolar surfaces, like plastics, but their adhesion also depends on their hydrophobicity [9][10].

Biofilms formed on low-shear surfaces have a low tensile strength and break easily, whereas biofilms formed on high-shear surfaces are remarkably strong and resistant to mechanical breakage. It appears that turbulent flow also enhances bacterial adhesion and biofilm formation [11].

As for the selection of materials for biofouling detection, polymeric materials have been preferred mainly polyvinyl chloride (PVC) and polyethylene (PE). Niquette et al. [12] showed that the biofilms growth on PVC and PE was inferior to bacteria growth on iron matrices, and also that the viable counts on these plastics were similar. Unlike, the research of Cloete et al. [13] revealed that the biofilm formation on PVC surfaces was superior than on stainless steel. Lehtola et al. [14] found that biofilms grew faster on PE than on copper pipes, and that the biofilm was less influenced by the surface material than by the chlorine content. Schwartz et al. [15] studied the biofilm development on high density polyethylene (HDPE), PVC, and copper. They concluded that the density of viable cells on these materials were 35 – 38 %, except for copper that was less than 10 %.

Microorganisms contaminate all kind of surfaces, and given bacteria may adhere on a surface for months. In hospitals, an effective cleaning and the use of copper (antibacterial agent) surfaces is an approach to aid current hygiene practices [16].

Commercial coupons of stainless steel, carbon steel and copper are available in the market for corrosion monitoring [17]. Likewise, commercial biocoupons for the detection of biofouling formation, are also available in the market although they are extremely expensive, Fig. 2 [18].

![Fig. 2 – Biocoupons for the potential of biofilm formation determination [18].](image)

In this work, the materials selected for the plane coupons that will be tested in alternative of the biocoupons in the market, were two of the classic ones used for corrosion detection, i.e., copper and stainless steel, two plastics, namely, PVC and HDPE, besides neoprene which is a synthetic rubber that favours the biofilm growth due to its fibrous surface. The cylindrical coupons were in stainless steel and PVC, analogously to the DSS sensor tubes.

2. Experimental

2.1. Laboratory set-up

In order to assess the performance of the several materials selected for the biofouling detection, a laboratory set-up was mounted (Fig. 3).

The test fluid was recirculated in closed mode by a centrifugal pump (Grundus UPS 25 – 60 N), that pumped it from reservoir 1 (V = 25 L) to two parallel pipelines of SS (D = 8 mm) and PVC (D = 10 mm). The fluid was heated by an electrical resistance (TETRA HT 100), the temperature ranging from 20 to 28 °C.

To test the performance of DSS sensor tubes and cylindrical coupons both of SS (D = 8 mm) and...
PVC (D = 10 mm), the flow rates were duly set such that the average crossflow velocity in DSS sensor tubes and in the cylindrical coupons was 0.5 m·s⁻¹, the Reynolds numbers therein being somewhat similar.

As shown in Fig. 3, in each pipeline there was a DSS sensor tube that monitored the vibration amplitude and four cylindrical coupons to quantify the mass of adhered biofilm.

Simultaneously, five plane coupons of copper, stainless steel, HDPE, PVC and neoprene were tested for biofouling detection. An aquarium pump pumped the fluid test with a flow rate of 35 L·h⁻¹ into reservoir 2 (V = 6 L) where the plane coupons were located. Reservoir 2 had no heating and the heat was dissipated by natural convection, thus the temperature in reservoir 2 was lower than in reservoir 1.

Table 1 and Fig. 4 present the cylindrical coupons dimensions and a picture of these coupons, respectively.

### Table 1 – Dimensions of the cylindrical coupons.

<table>
<thead>
<tr>
<th></th>
<th>L (mm)</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>SS</td>
<td>8</td>
<td>47</td>
<td></td>
</tr>
<tr>
<td>PVC</td>
<td>10</td>
<td>59</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 3 – Laboratory set-up.

Fig. 4 – Cylindrical coupons: a) SS, b) PVC.

Table 2 and Fig. 5 present the plane coupons dimensions and a picture of these coupons, respectively.

### Table 2 – Dimensions of the plane coupons.

<table>
<thead>
<tr>
<th></th>
<th>L₁ (mm)</th>
<th>L₂ (mm)</th>
<th>L₃ (mm)</th>
<th>D (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>76</td>
<td>13</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>SS</td>
<td>76</td>
<td>12</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>PVC</td>
<td>75</td>
<td>12</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>HDPE</td>
<td>75</td>
<td>12</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>Neoprene</td>
<td>76</td>
<td>13</td>
<td>8</td>
<td>-</td>
</tr>
</tbody>
</table>

Fig. 5 – Plane coupons for biofouling detection: a) copper, b) SS, c) HDPE, d) PVC, e) neoprene.

2.1.1. Diveil Surface Sensor (DSS)

The Diveil Surface Sensor (DSS) is a device marketed by Enkrott, S.A., to detect and evaluate the quantity and nature of the deposits adhered on a material surface [19]. The adhesion, growth and removal of deposits produce variations on the vibration amplitude of a monitored surface. The DSS comprises a sensor and an actuator that are attached to the outer surfaces of DSS sensor tubes. The actuator forces the sensor tube to vibrate and the vibration response is captured by the sensor. Both actuation and sensing processes are performed automatically at constant time intervals [19].

In industrial applications, the customer may choose one of the methods for detection of biofouling, DSS and / or coupons, depending on the analysis speed, contaminations and costs of biofouling monitoring.

2.2. Test Fluid

The test fluid consisted of a water sample collected from a cooling water system from a food industry. The characterization of this water is listed in Table 3, namely, pH (Hanna Instruments TPM –
99121), conductivity – k (Hanna Instruments HI – 8733), viable bacterial count and bacterial summary identification.

Table 3 – Test fluid characterization, before and after the biofilm adhesion assay.

<table>
<thead>
<tr>
<th>pH</th>
<th>k (µS/cm)</th>
<th>Viable count (CFU × 10^8/100 mL)</th>
<th>Bacterial summary identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>6,5</td>
<td>1580</td>
<td>0,67</td>
<td>20 % positive coccus</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>80 % negative coccus</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>24 % positive coccus</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>76 % negative coccus</td>
</tr>
</tbody>
</table>

2.3. Experimental procedures

For the characterization of the plane coupons materials and for the biofouling adhesion / removal assay the following protocols / procedures were used.

2.3.1. SEM Microscopy

The Scanning Electron Microscopy (SEM) is used for metallurgic I&D and quality control. SEM emits electron beams which interact with the sample to create an image of its surface topography. It visualizes surface pitting, deposits, contaminants, particles, etc. [20].

For this method, the plane coupons of copper, stainless steel, PVC, HDPE, and neoprene were cut and washed with alcohol to remove grease. For the metallic coupons, conductive tape was used to fix the samples on the sample holder and guarantee electron conduction, as well. For the plastic coupons and neoprene, besides the conductive tape, they were coated with a thin layer of a conducting material (chromium), using a sputter coater (QUORUM – Q150TES).

After these pre-treatments, the samples were inserted in a SEM microscope (JEOL JSM – 7001F) and visualized.

2.3.2. Goniometry

The contact angle is related to the surface energy, roughness and heterogeneity [21].

For this method, the samples of copper, stainless steel, PVC, and HDPE were cut, and dried in a vacuum furnace (Lab Line Duo-Vac Oven, P(abs) = 5 cm Hg). The contact angles determinations were carried out using a microscope (Wild Heerbrugg M3Z), a focus light (Leica Type: MTR31), a sample chamber (Ramé Hart Inc, 1000700) and an analysis software (Axisymmetric)

Drop Shape Analysis – Profile: ADSA-P). Five drops were analysed for each material, and the results presented herein are the average of the threshold obtained for each drop.

2.3.3. Biofilm adhesion

Before each assay, reservoir 1 was filled with test fluid, by opening all the valves, turning on the pumps and setting the flow rates such that the average crossflow velocities were 0.5 m·s⁻¹ in all pipes. The system was monitored until no air was present. Afterwards, the acquisition software was turned on and set to take data every 30 min.

The system was automated, thus the daily procedure consisted only on saving the data acquired by the software on the fortnight, guaranteeing that there was no air in the system and controlling the flow rate in the two pipelines.

2.3.4. Cylindrical Coupons

The selection of the days for the withdrawal of the cylindrical coupons was made by analysing the DSS data and/or the inspection of the crystal (transparent) tubes. Obviously, the coupons withdrawals were an intrusion to the system.

The coupons were dried and weighted (Mettler Toledo AB204 analytical scale) after its removal, washed with detergent and water, and dried and weighted once again.

2.3.5. Plane Coupons

The plane coupons were withdrawn preferably on the same days of the cylindrical coupons removal. They were dried and weighted, washed with detergent and water, dried and weighted once more.

2.3.6. Biofilm removal

The cleaning in place (CIP) can either be mechanical or chemical. As the system conditions would vary by a mechanical cleaning, a chemical cleaning was performed, by using acid and basic solutions such that most deposits were eliminated.

Firstly, tap water circulated in open mode. Then, a solution of nitric acid (VWR – 30% of purity) was poured to reservoir 1, until pH 3 was reached, to remove scaling. After 30 min, the acid solution was drained and simultaneously tap water circulated, till a neutral pH was attained. To remove adhered organic and biological deposits, a solution
of caustic soda (Sodacasa – 99% purity) was poured to reservoir 1, until pH 14 was achieved. After 1 h, the basic solution was drained and simultaneously tap water circulated, till a neutral pH was attained.

3. Results and discussion

3.1. SEM Microscopy

The SEM images for all plane coupons were obtained through the procedure mentioned in section 2.3.1 (Fig. 6 – Fig. 9).

Fig. 6 – SEM image of copper (3000 magnification).

In Fig. 6, copper appeared to show a high surface roughness, most likely due to oxides and/or biofouling. The image suggests that the biofilm was not completely withdrawn by the successive washings.

Fig. 7 – SEM image of stainless steel (3000 magnification).

Analogously, the stainless steel also revealed high surface roughness due to the formation of oxides and/or biofilm deposits (Fig. 7).

Fig. 8 – SEM image of HDPE (3000 magnification).

The HDPE presented a smooth surface without any porous at all (Fig. 8).

Fig. 9 – SEM image of PVC (3000 magnification).

Likewise for the PVC, there was no significant surface roughness but there were some surface pores with sizes up to 10 μm (Fig. 9).

Fig. 10 – SEM image of neoprene (50 magnification).
Monitoring of Deposition in Water Conducts: Determination of which materials favour the Biofouling Formation in Cooling Water Systems

Neoprene revealed an upper surface with twisted strings and underneath it a spongiform structure with moderate porosity (Fig. 10).

3.2. Goniometry

Table 4 and Fig. 11 present the goniometry data and the pictures of the water drops on each material, respectively.

Table 4 – Contact angles for the various materials of the plane coupons.

<table>
<thead>
<tr>
<th>Material</th>
<th>Contact Angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>84 ± 3</td>
</tr>
<tr>
<td>SS</td>
<td>68 ± 2</td>
</tr>
<tr>
<td>HDPE</td>
<td>68 ± 2</td>
</tr>
<tr>
<td>PVC</td>
<td>67 ± 2</td>
</tr>
</tbody>
</table>

Fig. 11 – Pictures of the water drops throughout the contact angles assays: a) copper, b) SS, c) HDPE, d) PVC.

3.3. Biofouling Monitoring

A normalization of the vibration amplitudes registered by the Diveil Surface Sensor, was done by subtracting the initial vibration amplitude (t = 0) to the actual amplitude. Furthermore, a proper correction to the temperature of 25 °C was done, the final result being named $Amp_{NC}$.

3.3.1. Biofilm adhesion on DSS sensor tubes

For 36 days, the DSS acquired the evolution of the vibrations amplitude, as depicted in Fig. 12.

The vibration amplitude of the SS sensor tube presented a sharp development until day 6, a plateau till day 14, followed by a slow decrease up to day 29 and on day 30 it vanished till the end of the assay, in close agreement with the biofilm development theory.

As for the PVC sensor tube, a late development of biofilm was observed, only on day 29, and the maturation took place on day 33. Meaning that the PVC sensor tube is less sensitive to biofilm development.

3.3.2. Biofilm adhesion on cylindrical coupons

Fig. 13 depicts the biofilm dry mass per surface area unit for the cylindrical coupons of SS and PVC.

Similarly to Fig. 12, there was a considerable growth of adhered biofilm on both materials on the days 3 and 31, for SS and PVC, respectively. Between the days 31 and 36, the biofilm on the SS coupon matured and the cells on the outer layers migrated, to form biofilms elsewhere, thus the biofilm mass decreased.

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1 NC stands for normalized and corrected to 25 °C
The vibration amplitude of the DSS sensor tubes showed a linear relationship with the adhered biofilm mass (Fig. 14), in agreement with the work of Pereira [22].

Despite the poor correlation coefficient, both materials imply a linear relationship between the vibration amplitude and the biofilm mass, as expected. Surprisingly, the ratio of the slopes of the relationships is practically 1, and the relationship between $Amp_{NC}$ and the biofilm mass is unique, regardless the material.

Assuming the density and the viscosity of water at 25°C for the test fluid and using the definitions of Reynolds number and surface shear stress and also the Moody’s diagram, the Reynolds number, friction factor ($f$) and surface shear stress ($\tau_o$) were calculated.

$$\tau_o = \rho \frac{v^2}{R}$$

Tab. 1 – Friction factor and shear stress for the cylindrical coupons.

<table>
<thead>
<tr>
<th>Material</th>
<th>$D$ (mm)</th>
<th>$\varepsilon$ [23] (mm)</th>
<th>$Re \times 10^{-3}$</th>
<th>$f$</th>
<th>$\tau_o$ (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS</td>
<td>8</td>
<td>45</td>
<td>4.5</td>
<td>0.011</td>
<td>1.37</td>
</tr>
<tr>
<td>PVC</td>
<td>10</td>
<td>1.5</td>
<td>5.6</td>
<td>0.009</td>
<td>1.12</td>
</tr>
</tbody>
</table>

In fact, the mass of the adhered biofilm was generally higher on SS coupons than on PVC coupons, i.e., $m_{SS} > m_{PVC}$, matching the surface shear stress order, $\tau_o_{SS} > \tau_o_{PVC}$, in agreement with the data obtained by Donlan and Costerton [11]. Although the surface shear stress on SS coupons is only 22 % higher than on PVC coupons, this shear stress gap corresponds to ca. 50 % more biofilm mass on the stainless steel.

Therefore, the surface shear stress (including the relative roughness, the inside diameter and the average crossflow velocity) is most likely the most relevant factor in the selection of tubular materials for the biofilm detection in water piping.

### 3.3.3. Biofilm adhesion on plane coupons

Fig. 15 and Fig. 16 depict the dry mass of adhered biofilm deposited on the plane coupons, with and without neoprene, respectively.

By far, neoprene presented the highest biofilm mass, in agreement with the SEM image of this material (Fig. 10) that showed plenty of irregularities on this material surface. In fact, microorganisms are prone to adhere to the irregularities, as mentioned by Grenho [9]. As for the other plane coupons, $m_{SS} > m_{PVC} = m_{HDPE} > m_{copper}$ (Fig. 16), similarly to the observations of Niquette et al. [12] for iron, PVC and PE.

For the stainless steel, it was clear the growth and development of the adhered biofilm mass until day 31, and in between the days 31 and 36 there was a decline, pointing out that the biofilm matured and the outer layers migrated. This phenomenon may also be caused by the loss of biofilm in the coupon removal due to the biofilm slippery texture in the hydrated stage.

Although the response of SS plane coupons was substantial, they are not recommendable for biofilm detection because they are prone to corrosion.
The significant biofilm mass on SS coupons is probably due to its high absolute roughness, 45 μm, i.e., 30-fold highest than the other materials (PVC, HDPE and copper) with 1,5 μm. This fact was corroborated by the SEM images in Fig. 6, Fig. 7, Fig. 8 and Fig. 9 (except for copper).

As for the polymeric plane coupons of HDPE and PVC, they presented substantial masses of biofilm adhered, thus they may be used for biofilm detection, especially because they are corrosion-free.

HDPE and PVC coupons, with equal absolute roughnesses and similar contact angles, 68° and 67°, respectively, presented very similar biofilm masses, as expected.

As for the copper, it was expected a much higher mass for two distinct reasons: it was the most hydrophobic material (84°), which would favour the microorganisms adhesion; and the SEM image (Fig. 6), it showed a high effective roughness. Still, the biofilm mass on the copper coupons was the lowest maybe due to the copper bactericide effect \(^{[16]}\) and / or defective washing after the adhesion assay. Nevertheless, the unexpected results of the copper coupons are analogous to the data reported by Lehtola et al. \(^{[14]}\) and Schwartz et al. \(^{[15]}\).

Thus, it was concluded that the effective roughness and the hydrophobicity are probably the most relevant factors in the selection of plane materials for the detection of biofouling in industrial processes, as reported by Grenho \(^{[9]}\). In fact, the order of the adhered biofilm masses on the coupons based solely on the effective roughness and the hydrophobicity would be: \(m_{neoprene} \gg m_{copper} \approx m_{SS} > m_{PVC} \approx m_{HDPE}\), which corresponds to the reality, except for the copper.

Despite the distinct temperature, pressure (pipe lines and reservoir 2), geometry (cylindrical and plane) and average crossflow velocity (pipe lines and reservoir 2), the data of the plane and cylindrical coupons of SS (Fig. 17) and PVC (Fig. 18) were compared.

![Fig. 17 – Dry mass of biofilm per surface area unit of the cylindrical and plane SS coupons.](image)

The adhered biofilm mass on SS coupons was similar in the two geometries, in spite of the distinct operating conditions. Up to day 30, the development of biofilm was favoured on the cylindrical geometry. From day 31 till the end of the assay, the plane coupons favoured the biofilm growth.

![Fig. 18 – Dry mass of biofilm per surface area unit of the cylindrical and plane PVC coupons.](image)

The PVC plane coupons favoured the biofilm development up to day 23, whereas the PVC cylindrical coupons showed a higher biofilm growth thereafter.

In sum, the cylindrical coupons, especially the ones in SS, promoted a highest biofilm adhesion and development, meaning a more effective biofouling detection. Yet, this benefit may not only relate to the geometry since the temperature and the average crossflow velocity in the pipes were higher than in reservoir 2, also favouring the biofilm adhesion.

### 3.3.4. Biofilm removal from the DSS sensor tubes

At the end of the adhesion assay on the DSS sensor tubes, a cleaning in place (CIP) was performed, to test the ability of the materials response to the removal of adhered biofilm from the pipes.

![Fig. 19 – Cleaning in place of the stainless steel DSS sensor tube.](image)
Throughout the CIP assay, Fig. 19, immediately after each and every fluid exchange the vibration amplitude of the SS sensor tubes revealed a steep scattering. After a stabilization period, the signal vanished, indicating an effective removal of the deposits and the CIP ended.

![CIP - PVC](image)

**Fig. 20 – Cleaning in place of the PVC DSS sensor tube.**

As for the PVC DSS sensor tubes, Fig. 20, the variations of the vibration amplitude were also noticed, although data scattering was lower than for the SS sensor tube.

The scattering of the vibration amplitudes of each sensor tube whenever an exchange of fluids took place, was due to:

- Simultaneous air inlet with the cleaning fluids (tap water, acid solution (HNO₃) and basic solution (NaOH));
- Formation of micro bubbles when the acid solution was poured;
- Biofouling swelling, i.e., the biofilm swelled and contracted, whenever the operating conditions were severe [24].

4. Conclusions

The goal of selecting and testing materials that favour the biofilm formation and development on cylindrical and plane coupons, and on the Diveil Surface Sensor (DSS) tubes, for the ease and early detection of biofouling adhesion, was fulfilled.

One assay of adhesion / removal on DSS sensor tubes of SS and PVC, on cylindrical coupons of the same materials and diameters, and on plane coupons of copper, SS, HDPE, PVC and neoprene were carried out. The plane coupons materials were characterized by SEM microscopy and goniometry.

DSS sensor tubes of SS and PVC revealed to be suitable for the detection of biofilm adhesion / removal, the SS being more effective. For the cylindrical coupons, with the same material and diameter as the DSS sensor tubes, the same conclusion was drawn, the SS coupons being the ones where a higher mass of biofilm adhered at a higher rate. The preferential adhesion of biofilm on SS is most probably due to the higher surface shear stress on this coupon, in comparison to PVC.

The ratio of the slopes of the straight lines of the vibration amplitude on the DSS sensor tubes vs. dry mass of adhered biofilm is practically unitary, indicating a unique linear relationship, irrespective to the material.

From the plane coupons developed for early detection of biofilm, the neoprene coupons yielded the highest biofilm adhesion. The response of SS plane coupons was also very significant, although they are not recommendable for the biofilm detection because they are prone to corrosion. On the other hand, the polymeric plane coupons of HDPE and PVC presented substantial masses of biofilm adhered, thus they may be used for biofilm detection, especially because they are corrosion-free. The lowest mass of biofilm adhered was found for the copper coupons, therefore their use is definitely not recommended for this purpose. For the plane coupons, the effective roughness and the hydrophobicity are most likely the most relevant factors in the selection of plane materials for the detection of biofouling.

In general, the cylindrical coupons promoted a higher adhesion and development of biofilm compared to the plane coupons of the same materials, and therefore a more effective detection of biofouling. However, it is impossible to assign this benefit only to the cylindrical geometry since the temperature and the average crossflow velocity in the pipes are higher, hence also promoting this phenomenon.

The lab set-up cleaning consisted of successive washings with a HNO₃ solution at pH 3 (for dissolving the scaling) and a NaOH solution at pH 14 (for the biofilm removal), with water washings in between. The vibration amplitude of the DSS sensor tubes revealed strong scattering after each and every solution exchange, finally stabilizing and vanishing by the end of the third water washing.

Acknowledgements

This work was accomplished in former Enkrott Química, S. A., and in Instituto Superior Técnico, for over a year, reflecting unforgettable and enriching knowledge. Professors Patrícia Almeida de Carvalho and Benilde Saramago are deeply acknowledged for allowing the SEM and Goniometry analysis.
Symbol List

- $\varepsilon$ Absolute roughness (m)
- $\rho$ Density (kg·m⁻³)
- $\mu$ Viscosity (Pa·s)
- $\tau_0$ Surface shear stress (Pa)
- $D$ Internal diameter (m)
- $L$ Length (m)
- $L_1, L_2, L_3$ Plane coupons dimensions (m)
- $T$ Temperature (°C)
- $v$ Average crossflow velocity (m·s⁻¹)
- $P$ Pressure (cm Hg)
- $Re$ Reynolds number

References
